

INDUSTRIAL MINERALS  
AND ROCKS

**The Seeley W. Mudd Series**





Seeley Wintersmith Mudd

*(Frontispiece.)*

# INDUSTRIAL MINERALS AND ROCKS

(Nonmetallics Other than Fuels)

First Edition

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## INTRODUCTION

**T**O the title of this volume—Industrial Minerals and Rocks—is added a highly important subtitle: Nonmetallics Other than Fuels. As an alternative might be substituted the qualifying phrase “except metalliferous ores and mineral fuels.” Native copper and petroleum, for instance, are industrial minerals, but they are not included in this book. Each of its forty-eight chapters deals either with a single or with a related group of those useful minerals or rocks commonly known as “nonmetallic.” Of these no less than 70 are mined and used on a substantial scale. A calculation based on an estimate of the world’s consumption, at average prices prevailing in the United States, indicates an annual production valued at between \$1,500,000,000 and \$2,000,000,000 at the mine. The United States alone accounts for more than half of the total; and the tonnage of such “ores” exceeds that of metalliferous ores, including iron, by a considerable margin.

Here is not only a large but a particularly fertile field for the engineer and technologist in the mineral industries. Only in recent years, and then only to a limited extent, have the search for, and the exploitation and beneficiation of, these minerals been conducted by men having professional engineering training. But more and more those who finance these operations are realizing the advantages of employing such men to manage and superintend their operations. The gradual exhaustion of surficial deposits and the consequent more elaborate mining methods; the need for mining ores of lower grade, resulting in the introduction of mechanical dressing and other types of processing; and the competition between materials, which demands that these things be done with the maximum efficiency—all these things require the highest possible skill, knowledge, and resourcefulness. The trend is distinctly toward the employment of technically educated men.

While many of the problems are essentially the same as those encountered in the metal and coal industries, there is enough of a special nature to warrant the introduction in “mineral industry” schools of sundry courses designed particularly for training in the nonmetallic industries. To assist undergraduate students and junior engineers to take advantage of the growing opportunity in this broad field is one of the purposes of this volume. And this is one of the considerations that moved the Seeley W. Mudd Memorial Fund Committee to sponsor it and recommend the appropriation from the Fund that made publication possible. Colonel Mudd was particularly mindful of the well-being of younger men in the

profession; and in the deed of gift special mention is made of projects that "will be of particular benefit to young engineers." It is believed by the Mudd Committee, consisting of Messrs. Harvey S. Mudd, Edgar Rickard, and George Otis Smith, that the volume will serve this purpose most effectively.

Samuel H. Dolbear first saw the need and developed the plan of publication when he became, early in 1935, the first Chairman of the newly established Industrial Minerals Division. Though he accepted chairmanship of the General Committee with real reluctance, having once undertaken the work he has devoted to the volume a tremendous amount of time and energy. To Oliver Bowles, Vice-chairman, goes much credit for his assistance in organizing the work; and to Heinrich Ries and George Otis Smith special acknowledgment is due for reading critically almost all of the manuscripts and suggesting important amendment and revision of many of them. The Institute likewise is indebted to the other members of the Editorial Board and of the Advisory Committee, as well as to the forty-seven individual contributors.

The U. S. Geological Survey, the Canadian Department of Mines and particularly the U. S. Bureau of Mines have been extremely helpful. Authors of many of the chapters are on the staff of the Bureau at Washington; and many of the other authors obtained invaluable statistics and technical data from the Bureau's files and publications.

Each chapter is written specifically for this volume by a man recognized as a specialist on his subject. Though the chapters were prepared independently, the assignments were so carefully made, the pattern was so painstakingly prepared, and the work was so skillfully coordinated by Mr. Dolbear and the Editorial Board that the volume is far more than a mere aggregate of disconnected papers. There is practically no duplication and there are few omissions of consequence; for a book of this character, the volume has unity and coherence in unusual degree.

We know of no book yet published for which any claim is made that it rivals this as a comprehensive, accurate, authoritative, and up-to-date treatise in its particular field—a field of rapidly increasing importance to the Institute. To the owners of nonmetallic mineral deposits and to those who purchase, process, and fabricate these minerals and who market the resulting products and manufactures, it is hoped the volume will prove useful. Certainly, it should be of great value to the growing group of industrial mineral technologists and engineers; and, as Colonel Mudd surely would have wished, among its greatest beneficiaries should be the young men of the profession.

A. B. PARSONS,  
*Secretary A.I.M.E.*

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INDUSTRIAL MINERALS  
AND ROCKS





# INDUSTRIAL MINERALS AND ROCKS

## (NONMETALLICS OTHER THAN FUELS)

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### CHAPTER I

### ABRASIVES

BY V. L. EARDLEY-WILMOT\*

**A**BRASIVES may be divided into two general classes, natural and manufactured. The former includes all rocks and minerals used for abrasive purposes without chemical or physical change, other than crushing, concentrating, shaping or bonding into suitable forms. Manufactured or artificial abrasives are made either by heat or chemical action from metals or raw mineral materials, and in recent years they have largely replaced natural abrasives, particularly for grinding metal.

Natural abrasives are found in many parts of the world. Roughly, the list includes all minerals capable of abrasive action, but the use of many of these as an abrasive is insignificant compared to their other uses. The principal natural abrasives in order of their hardness are diamond, corundum, emery, garnet, which are termed for convenience "high-grade natural abrasives," while "other natural abrasives" include mainly the various forms of silica, the chief of which is sandstone, which are listed separately under the heading of "Siliceous Abrasives."

Although abrasive materials are used in their natural, shaped or ground forms, they are mainly employed after fashioning into products such as grinding wheels and as abrasive-coated papers or cloths. Most of the wheels are made of manufactured abrasives, the quantity of emery and of corundum wheels being comparatively small. Grinding wheels are made by several processes, the products being known as: (1) vitrified, in which the bond is mainly clay; (2) silicate, having sodium silicate as the bond; (3) elastic, the bond being rubber; (4) those having Bakelite or artificial resin for the bond. Each form of wheel is made in many combinations, used for different kinds of grinding. In making the coated abrasive products, the sized grains of quartz, emery and garnet,

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or the manufactured silicon carbide and fused alumina, are automatically fed onto the moving glued backing of paper or cloth and after drying are covered with a thin coat of glue. The even distribution of the grain, both for "close coating" and "open coating," as well as the quality and temperature of the glue, are highly important factors requiring careful control. References to the methods of manufacture of these and other abrasive products will be found in the bibliography at the end of this chapter.

There is hardly an industry in which abrasives do not play some part, therefore the consumption of abrasives and abrasive products is largely dependent upon general business conditions. The automobile industry is the largest user, so that the total volume sales of abrasive somewhat closely precedes and parallels the fluctuations of the automobile output.

### HIGH-GRADE NATURAL ABRASIVES

#### INDUSTRIAL DIAMONDS

Diamonds are reviewed in detail in chapter XVI, therefore only their uses as abrasive are outlined briefly below. There are two kinds of abrasive or industrial diamonds, the black diamond called "carbonado" or carbon, and "bort," the latter including badly colored, flawed or broken fragments of diamonds, unsuitable for gems, which are obtained principally from South Africa.

Carbons come mainly from the State of Bahia, Brazil. They are the toughest and hardest of all diamonds and are used nearly always in the crowns of diamond drills. The application of diamond drilling in the search for oil and ores (especially gold) has grown very much in recent years and the consumption of carbons has increased accordingly. An important use is for diamond dies in the wire-drawing industry, and another in the tools used for dressing and truing grinding wheels. Diamond-tipped tools are used for turning and boring substances such as hard rubber, vulcanite, Bakelite or hard metals, and in such industries as the electrical and automobile, which demand very accurate grinding work. Diamond-toothed saws are extensively used for cutting various kinds of stone. Composition diamond grinding wheels, introduced comparatively recently, are meeting with success.

Diamond dust is made by crushing diamonds in a special airtight steel mortar and then grading by the rates of settlement in olive oil. The prepared dust is used mainly for grinding or lapping diamonds and other gems, hard minerals, rocks, and cemented carbide products.

During the World War the uses for industrial diamonds, particularly in munition factories, developed considerably. It has been estimated that in recent years the consumption for abrasive purposes amounts to about 40 per cent in quantity and between 15 and 20 per cent in value of

the world's output of diamonds of all kinds.<sup>3</sup> During 1935 the stimulating effect caused by the increased demand for armaments and the desire to replenish diminished stocks led to a greatly increased consumption of industrial diamonds, which for some uses, such as tools for dressing wheels, amounted to almost 90 per cent.

### CORUNDUM

Corundum, an oxide of aluminum, is, next to the diamond, the hardest mineral known. Only within the last century has corundum, in the form of grain, been commercially used for abrasive purposes, though the natural crystals may have been used by the ancient Egyptians for carving hieroglyphics on their stone monuments. In ancient times the only known use for corundum was as jewels, ruby and sapphire being transparent forms of the finest corundum, though they were not recognized as forms of that mineral until 1805. Emery, on the other hand, a mixture of corundum and iron oxides, has been used as an abrasive for several centuries. A little over a century ago true corundum was recognized in the United States from samples found in South Carolina, but corundum mining did not start until 1871.

### COMPOSITION

Corundum is the sesquioxide of aluminum ( $\text{Al}_2\text{O}_3$ ) and has a theoretical composition of 52.9 per cent aluminum and 47.1 per cent oxygen, although invariably it contains small amounts of impurities such as silica, iron, lime and water. (See table of analyses of emery.) It is practically unaffected by even the strongest acids.

Corundum is generally classified in three groups: (1) gem stones, (2) common corundum, (3) emery. The gem stones include the transparent varieties recognized by name according to their color, thus the red is ruby, blue is sapphire and the yellow and green are sometimes erroneously called topaz and emerald. (The true topaz and emerald are really silicates of aluminum and beryllium.) Common corundum is opaque and varies in color and shades. It occurs as crystals—as “block” corundum, a massive form showing but little development of parting planes, or as “sand” corundum, minute crystals or small irregular grains sometimes found in ore bodies occurring on the contacts of schists and gneisses. Emery is described in detail later.

### PROPERTIES

*Color.*—Corundum shades from the deep blues and reds of the gems into opaque dull hues through white, gray, pink, bluish to brown or bronze, the browns and bronzes being the commonest. Often the shading of individual crystals changes from the center to the outside.

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<sup>3</sup> References are at the end of the chapter.

*Hardness.*—The hardness is 9 on Mohs' scale. The various corundums differ in hardness, depending on the purity of the mineral, sapphire being the hardest.

*Crystal System.*—The system is a rhombohedral division of the hexagonal system, usually as long six-sided prisms that taper towards either end from the center, also short and stubby crystals assuming a barrel-shaped outline. The ends of the crystals are striated, forming equilateral triangles and the faces show distinct crosshatching. The common lamellar structure is due to twinning.

*Cleavage.*—Gem varieties break with a conchoidal fracture, but common corundum has a prominent basal parting causing crystals to break readily with smooth flat surfaces.

*Specific Gravity* varies from 4.10 to 3.93, depending on the amount of impurities present.

*Other Properties.*—Luster is adamantine to vitreous; sometimes pearly. It is transparent to almost opaque, depending on impurities present. It is very permeable to X-rays, which serves to distinguish the true gems from artificial stones as well as from minerals such as garnet or colored quartz. Pleochroism is very strong. The refractive index is high, but there is a low double refraction.

#### ORIGIN AND MODE OF OCCURRENCE

Corundum is known to occur in about 20 kinds of rocks and in about 35 different countries throughout the world. The mineral is usually associated with peridotite in the United States, nepheline syenites in Canada and feldspathic rocks in India and South Africa. The origin of corundum has been discussed in some detail by several authors, among whom are A. E. Barlow,<sup>1</sup> J. H. Pratt,<sup>10</sup> A. L. Hall, T. H. Holland and others, a summary of each being given in the Mines Branch, Canada, report.<sup>2</sup> It appears generally accepted that corundum was one of the first constituents to crystallize out from the molten magma and, further, that in certain supersaturated aluminosilicate magmas the whole of the excess of the alumina separates out as corundum, provided that the iron, magnesia and silica present are low, but if these are high it separates out as spinel, cordierite and other minerals (ref. 2, p. 24). Corundum consequently does not occur in the immediate vicinity of free silica, such as quartz.

The South African corundum is invariably associated with pegmatite intrusions into basic rocks.<sup>4</sup>

#### DISTRIBUTION OF DEPOSITS

Many of the occurrences of corundum are only of scientific interest. The countries having the main deposits of commercial importance

are the United States, Canada, Union of South Africa, India, Madagascar and Russia.

### *United States*

Corundum was first recognized in the United States a little over a century ago from a specimen submitted for identification from the Laurens district, South Carolina. Systematic mining for corundum, as distinguished from emery and the gem varieties, was first started at Corundum Hill, North Carolina.

The mineral has been found in many localities throughout the Appalachian belt from Massachusetts to Alabama, but only a few deposits have been developed. The best deposits are found in Jackson, Macon, and Clay Counties, North Carolina; and Rabun County, Georgia. Between 1900 and 1905 the corundum deposits of Gallatin County, Montana, were also worked to a small extent.

The Corundum Hill mine, Macon County, North Carolina, is about 8 miles northeast of Franklin on Cullasagee Creek, and is the largest corundum mine in the United States. The mineral occurs in a peridotite rock near its contact with gneiss. The deposit consists of a blunt lens-shaped mass of dunite, about 1200 ft. long and 450 ft. wide, and covers an area of about 10 acres. A number of veins of high-grade corundum rock in the dunite were worked by open cuts and tunnels, but with one exception they all pinched out. From the beginning of operations until 1900 the mine produced annually 200 to 300 tons of cleaned corundum. Several other mines in the immediate vicinity and in the same formation have also been worked for corundum between 1880 and 1890; these include the Behr mine, which had the best equipped corundum mill in the state; the Isbel mine, where the mineral occurs in an amphibolite dike, and the Herbert mine, where a number of very small veins of almost solid corundum occur in peridotite.

The most important Jackson County deposits are in the extreme northeastern part, in the vicinity of Sapphire, where there are numerous outcrops of peridotite in which the corundum is associated with garnet.

All the Georgia corundum deposits occur in lime-soda feldspar veins intersecting basic magnesian rocks of the peridotite type, usually on a contact with mica schist in close proximity to hornblende gneiss. Scattered occurrences are known in 13 counties, but the Lucas mine at Laurel Creek, Rabun County, is one of the best known corundum mines in the United States and was successfully operated between 1880 and 1893. Massive pieces of corundum weighing several hundreds of pounds were frequently encountered. The mineral occurs in an oval mass of altered peridotite about 2000 ft. long and 800 ft. wide, and is enclosed in a foliated quartz-mica gneiss.

Several hundred tons of corundum were mined prior to 1903 from the vicinity of Elk Creek in the central part of Gallatin County, Montana, where the ore occurs in syenites and pegmatites. A small production was obtained from York County, South Carolina.

### *World*

*Canada.*—Corundum was discovered in Canada in 1876 in Raglan Township, Renfrew County, Ontario. There are three recognized belts. The main belt of corundum-bearing nepheline syenites is approximately 103 miles long, with some interruptions, with a maximum width of 6 miles stretching through Renfrew, Hastings and Haliburton Counties, Ontario. The middle belt, in Peterborough County, 25 miles to the south, is a club-shaped area about 8 miles long and  $1\frac{1}{2}$  miles wide at the head. The southern belt, in Frontenac and Lanark Counties, 65 miles east of the middle belt, is about 12 miles long by 2 miles wide.

Prospecting has been carried out in about 50 different localities, but the principal operations were at Craigmont, in Raglan Township, at the Burgess mine, in Carlow County, and Jewellville mine, in Radcliffe Township, all in the main belt. A small tonnage was produced from Methuen Township in the middle belt. The Canadian mineral is brown to bronze, but an interesting deposit of blue corundum occurs on the York River in Dungannon Township.

Full details of the Canadian corundum deposits and operations are given in the Department of Mines reports.<sup>2</sup>

*Union of South Africa.*—Except for small quantities from Namaqualand and Rhodesia, the Transvaal has supplied the bulk of the corundum produced from South Africa since 1912, when the first shipments were made. The corundum-bearing areas cover 3000 sq. miles in the Zoutpansberg-Pietersburg-Leydsdorp districts of northeastern Transvaal. The ore occurs: (1) as primary deposits in altered feldspar, known as "boulder" corundum, (2) as secondary or eluvial deposits resulting from weathering of the softer rocks, leaving loose corundum concentrated to about 10 to 15 per cent of the volume, known as "crystal." The deposits are generally comparatively small, irregular in form, variable in corundum content and occur scattered over a very large area.

The rocks are feldspathic with dark mica as the principal accessory mineral, and are known locally as plumasites and marundites.

The recent bulletin issued by the Department of Mines of South Africa<sup>3</sup> gives full details of the deposits and operations.

*Southern Rhodesia.*—The principal deposits occur in eastern Mashonaland. About 2 miles southeast of Ruopapi, a number of corundum-bearing claims have been developed. The mineral is found in mica schist associated with granite, which underlies the corundum gravels. The

schist is decomposed to 60 ft., where crystals of an average size of  $1\frac{1}{2}$  in. occur. The annual output is very small.

*India.*—India is the home of the ruby and the sapphire, the principal ruby mines being about 100 miles north of Mandalay in Upper Burma. Corundum is widely scattered over Mysore State, and occurs in pegmatites, diorites, basalts, etc. In recent years operations have been confined to the Salem district of Madras, while previous to 1920 a small output was maintained from near Pipra, in Rewah State. There is still, and for many generations has been, a certain trade in Indian corundum used by the old armorer and lapidary, and even today the material is a regular item of trade in the bazaar cities of Delhi, Agra and Jaipur, where the lapidary still flourishes.

There does not appear to be any regular mining of the mineral but it is collected in a casual way by agriculturalists and cowherds who dispose of it through agents to the larger dealer in the big cities.

Details of the annual production from various districts are given in the records of the Geological Survey of India.<sup>3</sup>

*Madagascar.*—Corundum occurs in a soft, decomposed siliceous rock, which forms the so-called "red earth" that covers a large part of the surface of the island; sometimes to a depth of 60 ft. In places this earth has been washed from the summits and sides of hills, leaving the heavy minerals such as corundum, which are concentrated at the bottoms. Typical deposits occur in the southwest of Ambositra; also at Maovatanana and Betafo. The mineral is usually opaque and bluish in color, showing mica incrustations. Production has been steady since 1910, dwindling from a maximum of 800 tons in 1919 to 9 tons in 1931. Mining operations were renewed towards the end of 1935, when 12 tons was exported and production was maintained on a comparatively large scale in 1936.

*Soviet Russia.*—Small lenses of coarse-grained corundum occur in the western part of the Ilmen Mountains near the town of Kyshtym. The deposits were worked during the World War and considerable tonnage of the mineral has been mined in recent years from near Lake Tscherkakul, in the Bajan-Aul division of the Pavlodar district.

#### COMMERCIAL CONTROL

In spite of the severe competition from manufactured abrasives, particularly fused alumina, or artificial corundum, the output from South Africa in 1935 almost equaled the record of 1926, and the imports into the United States were also the highest since that year. The cheapness and comparative ease with which the South African crystals are obtained by washing the eluvial gravels bars competition from the United States, Canada, and other countries in which the mineral is mined from the solid rock and must undergo crushing and concentration at considerable



expense. The bulk of the South African crystal corundum is shipped to one or two grinders in the United States, where it is crushed and graded for the market.

Any large consumption of corundum in the future is unlikely because of the development of manufactured abrasives, with their highly controlled products, their ability to duplicate requirements exactly and consistently and their constant improvements and the expansion of their application. The extent of this all-round control is impossible with the natural mineral.

The maintenance of the market for South African corundum in the face of the competition from manufactured abrasive is due to its special suitability for certain uses, comparative cheapness and in recent years to the existence of efficient channels for its distribution in the United States, and to the fact that the grading of all corundum for export now comes under government control. Prior to the improvement of the grading and export regulations, some manufacturers, both in Europe and in the United States, had unfortunate experiences with regard to the quality and regularity of shipments. Efforts to gain the European market have failed, owing to lack of a proper distribution and selling organization as well as to loss in confidence from past irregular shipments, but early in 1936 an increased demand for corundum came from England.

#### PRODUCTION AND CONSUMPTION

Recorded statistics of true corundum production with the exception of Canada and South Africa are either intermittent or unobtainable.

##### *World*

*United States.*—Mining of corundum first started in North Carolina in 1871. Except for a little activity during the World War, there has been no production since 1905. This was first due to the keen competition from Canada, which started a little prior to that year, and since the beginning of the war, from South Africa. Apart from these sources of the natural mineral, the United States and Canada are the leading producers of highly efficient manufactured abrasives, a fact that does not encourage the production of natural corundum.

United States statistics group corundum and emery together, so that the true corundum output is not recorded except between 1894 and 1900, when separate records showed a total of 3720 tons of corundum.

*Canada.*—During the period 1900 to 1921, Canada was a regular and for many years the world's leading producer of true corundum. The peak was reached in 1906 and 1907, when 2500 and 3000 tons of graded grain were sold. During the last three years of production, the corundum was obtained as concentrate from treating the old tailing dumps. The total output during the 21 years amounted to 20,422 tons of graded grain

valued at \$2,104,251. There has been no production since 1921 other than an occasional shipment.

*Union of South Africa.*—Production has been steady since 1912, that of the peak year in 1926 being 5996 short tons and the next largest 4851 tons in 1936. The total shipments to date amount to 47,423 short tons valued at £341,420. At least 90 per cent of this has been in the form of crystal derived from eluvial deposits, recovered mainly by primitive methods. Owing, however, to the depletion of the known eluvial, more attention is now being paid to reef deposits and probably about half the present product is derived from reefs and in the future the proportion is likely to be even higher. The Union production since 1920 has been as follows:

YEAR	LONG TONS	YEAR	LONG TONS
1920	243	1928	1791
1921	110	1929	3220
1922	2035	1930	2275
1923	2928	1931	1008
1924	1288	1932	323
1925	1740	1933	1164
1926	5388	1934	2859
1927	917	1935	4264
		1936	4322

*Other Countries.*—Southern Rhodesian production from 1924 to 1930 was 37, 43, 39, nil, 22, 19 and in 1930 it was 10 long tons. Since then the output has ceased. Namaqualand produced 13 tons in 1920; 10 tons in 1923 and none since. India in 1920 and 1921 recorded 210 tons in 1920 and 64 tons in 1921 and from 1925 to 1930 it has produced 10, 28, 65, 21, 34 and 30 long tons, by years. Madagascar's output from 1920 to 1931 was as follows: 513, 280, 183, 411, 145, 222, 115, 150, 113, 113, nil and 1. The substantial output from Soviet Russia may include emery, which is also produced from the same region as that of corundum. The figures given by the Imperial Institute (London) Statistical Summary for Russia for 1924 to 1928 are 1409, 709, 884, 2636, and 6876 long tons.

#### MINING METHODS AND PREPARATION FOR MARKET

In most instances ordinary open-cast methods are employed in mining corundum. In the Transvaal most of the mineral is obtained by individual farmers or diggers, it being estimated that during the first half of 1935 over 300 Europeans and about 4000 natives were engaged in digging corundum. The openings are shallow, very few being as deep as 20 ft. At the deeper workings, which are down about 100 ft., as at Blinkwater, Borkum and Westphalia, the broken rock is hoisted by means of hand-operated or power-driven cranes, or shoveled up by stages from one platform to another.

In the eluvial deposits of the Transvaal, where the corundum is clean and generally free from adhering impurities, the material is first passed

over rocking inclined screens of about 1-in. mesh and thence over  $\frac{1}{8}$ -in. screens. The minus 1 plus  $\frac{1}{8}$ -in. material is washed in rotary pans and then hand-gravitated or jigged on small circular hand sieves. The large corundum crystals are picked out during the coarse-screening operations. The cleaned corundum is collected, sun-dried and graded into three sizes, as laid down in the Export and Grading Regulations, the final grading being done at the various buyers' depots. Boulder corundum, being in a massive form, is merely broken up by blasting and sledge hammers into fragments small enough to be handled for sale and export.

In the reef corundum or primary deposits, mining has been confined mainly to the friable and weathered plumasites or micaceous gneisses in which the corundum crystals break away with but little adhering gangue. The mineral is extracted first by hand-cobbing and the residue is cleaned by revolving in specially fitted cylindrical metal drums whereby the soft gangue is knocked off the crystals. At the Geber deposit at Blinkwater, which at present is the largest and most consistent producer in northern Transvaal, a better type of mill is used. The plus  $\frac{1}{8}$ -in. material is revolved at a high speed in a 10-ft. by 18-in. steel pipe through which a shaft with numerous beater blades is fitted, operated either wet or dry. It is more drastic and quicker than the barrel or other types. Grain corundum and concentrate are being produced by the Transvaal Corundum Co. near Bandolier Kop from the hard reef deposits. Ordinary wet gravity methods of crushing, stamping, jigging and tabling are employed. A flowsheet of this plant and much detail as to the treatment of South African corundum is given in the recent bulletin by W. Kupferburger.<sup>6</sup> References to treatment plants in Canada and the United States will be found in the bibliography at the end of the chapter.

The crude South African crystal corundum exported to the United States is ground, screened and graded into sizes required by the market. The sizes of the grain range from 6 to 200 mesh by screening and to F, or flour, grades by means of air and water classification.

#### SPECIFICATIONS, MARKETING, USES AND PRICES

*Specifications.*—For abrasive purposes corundum should have a bright and glassy luster and not be dull; the broken grain should be angular, uneven and sharp, not smooth or even; cleavage or parting planes should be absent, or nearly so; there should be no inclusions of other minerals; as a general rule the large corundum crystals are better than the very small ones.

*Marketing.*—The South African corundum is mainly collected by diggers and brought to central distribution agents. The Gifter Corundum Co. has recently erected large buildings for the treatment of corundum and has also established a depot in the heart of the corundum fields, so that diggers will be able to effect delivery within a few miles of the

producing sites. No corundum is exported without being certified by the government grading organization as up to the standards and specifications given in the Restricted Minerals Export Act No. 35 of 1927. Grade A is over 92 per cent  $\text{Al}_2\text{O}_3$ ; B is 90 to 92 per cent; C is 85 to 90 per cent and D is under 82 per cent  $\text{Al}_2\text{O}_3$ . Full details are given in the *South African Bulletin*,<sup>6</sup> and in the *South African Mining and Engineering Journal*.<sup>4</sup>

*Uses.*—The chief uses for corundum are as gem stones and abrasives. Gem, or precious, corundum is covered in chapter XVI.

The corundum of fine and flour grades is used as a loose grain for polishing rock specimens, gem polishing and for glass grinding and beveling, the largest consumption being in the latter trade, particularly for grinding lenses. At one time, before the introduction of manufactured abrasives, corundum was extensively used for "sand" papers but on the American continent is no longer employed for that purpose. The graded grain is mixed with bonds and made into grinding wheels, mainly by the vitrifying process. For certain purposes, some users prefer corundum to the manufactured abrasive wheels. About 70 per cent of the corundum used in the United States is made into grinding wheels and about 20 per cent goes to the glass and polishing trades.

In the United States a few hundred tons of South African corundum are used instead of bauxite for an alumina abrasive specially fused in the electric furnace. Before the expiration of the patent in which pure white alumina is used as the ingredient, appreciably more corundum was employed, but at present only one firm, owning the corundum patent, uses the natural mineral as the ingredient of a special alumina abrasive.

*Prices.*—The production value within South Africa for 1935 for boulder corundum was \$18.30; crystal, \$37.50 and concentrate, \$59 per short ton, whereas the import value of crude crystals in the United States was \$61 per short ton.

## EMERY

Emery is an intimate mixture of granular corundum and magnetite with some hematite. It takes its name from Cape Emeri, on the island of Naxos, in the Grecian archipelago, where it occurs in great abundance and from which it has been mined for several centuries.

## COMPOSITION

Mineralogically, emery is divided into three varieties: (1) true emery (2) spinel emery, (3) feldspathic emery. It is regarded as a mechanical admixture.

True emery is a mixture of corundum and magnetite, with or without hematite derived from the magnetite, such as the Greek and Turkish emery, and is usually of a reddish black tint.

Spinel emery is a mixture of spinel (pleonastehercynite) corundum and magnetite, the corundum being present in variable proportions and sometimes entirely lacking. It is usually a heavy, black, fine-grained aggregate with dark gray crystals of corundum appearing in the best varieties. The crystals often are cracked and considerably altered to hydrous mica. This kind is mined in New York and Virginia in the United States.

Feldspathic emery is similar to the spinel but contains in addition from 30 to 50 per cent plagioclase. Pure magnetite often is found in streaks within this mass.

There are at least three recognized grades of emery: Greek or Naxos, Turkish and American. Each one varies as to its chemical and physical properties.

TABLE 1.—*Analyses of Emery from Different Localities*

Locality	Alumina	Silica	Magnetic Iron Oxide	Lime	Magnesia	Water	Total
Greece:							
Naxos	62.64	4.90	31.41	0.45	0.06	1.04	100.50
Naxos	68.53	3.10	24.10	0.86		4.72	101.31
Naxos	57.69	6.36	30.87	0.89	0.20	3.99	100.00
Nicaria	75.12	6.88	13.06	0.72		3.10	98.88
Turkey:							
Kulah.	63.50	1.61	33.25	0.92		1.90	101.18
Gumuch	60.10	1.80	33.20	0.48		5.62	101.20
Samos.	70.10 <sup>a</sup>	4.00	22.21	0.62		2.10	99.03
U. S. A.:							
Virginia.	45.38	2.53	41.23	0.06	5.71	1.32	96.23
Chester, Mass.	50.02	3.25	44.11			n.d.	97.36
New York	59.22 <sup>b</sup>	0.84	30.68		3.54	2.70	96.98
New York	50.10	14.32	28.17	0.84	4.31	n.d.	97.74

<sup>a</sup> Also contains 3.72 per cent TiO<sub>2</sub> (ilmenite) and some of the alumina as spinel.

<sup>b</sup> Also contains 3.28 per cent TiO<sub>2</sub> (ilmenite) and some of the alumina as spinel.

### PROPERTIES

Emery is a massive, nearly opaque, dark gray to blue-black, sometimes mottled, mineral. Its specific gravity varies from 2.7 to 4.3; it has a hardness of about 8, breaks with a moderately regular fracture, and is always more or less magnetic. The appearance, hardness, composition and color vary according to the locality in which it occurs. The hardness depends upon the amount of corundum contained and the emery may be either fine grained or coarse grained, depending on the size of the corundum crystals present.

### ORIGIN AND MODE OF OCCURRENCE

Emery usually occurs in crystalline limestones and schists, also in peridotite and diorite. It is often associated with chlorite in hornblende

schists. Further details of the mode of occurrence are given in the description of world deposits.<sup>2,4,7,8</sup>

## DISTRIBUTION OF DEPOSITS

### *United States*

Emery is known to occur at a number of places in the eastern United States, but has been mined only from Chester, Mass.; Peekskill district, New York, and Whittles, Virginia.

*Massachusetts.*—Emery was discovered in Hampden County, at South Mountain, near Chester, in 1864 by Dr. H. S. Lucas, although 30 years previously the deposit had been unsuccessfully worked as an iron mine. Previous to 1913, practically all the emery from the United States was produced from this locality. The deposits are associated with a narrow band of amphibolite that extends almost continuously across the state. In the vicinity of Chester this band is about  $\frac{3}{4}$  mile wide, and the emery deposits can be traced for about 5 miles. The deposits occur in the form of a vein in the eastern side of the band, in close proximity to sericite schist. The emery is found in pockets from a few feet to 12 ft. wide and has been mined in places to a depth of 300 ft. Six or seven mines were operated along this vein and large quantities of ore were extracted, but the workings have been abandoned since 1913.

*Virginia.*—A spinel emery occurs to the west and south of Whittles, Pittsylvania County. Although the deposits were exploited many years ago for iron ore, they were not successfully worked for an abrasive until 1917 and for the next 11 years yielded a large quantity of good material.

There are two types of occurrence: (1) schist ores, in which thin bands of quartzite, interbedded with the schists in places, form one, and sometimes both, walls of the emery bodies; the rock being closely jointed and in many places pegmatitic; (2) granite ores, or those enclosed in a decayed granite, which often is cut by narrow, pegmatite dikes. The emery in both types is a heavy, black, fine-grained crystalline aggregate. Weathering has progressed to an advanced stage, so that in all the ore deposits the rocks are entirely decomposed, and owing to alteration of the schists into varicolored mottled clays, the surfaces of the emery bodies are often coated with a deep red clay. The ore bodies occur as irregularly shaped lenticular masses of solid emery, the largest of which is about 130 ft. long and 6 to 8 ft. wide.

The method of mining was based on the strongly developed joint structure of the emery bodies. After the decomposed rocks were removed, the emery blocks were dislodged by forcing iron bars into the joints. Very little dynamite was used.

*New York.*—Emery deposits of the spinel variety occur east of Peekskill, Westchester County, and have been operated by various

companies since 1883.<sup>5</sup> The ore occurs on the border of an igneous complex composed of hornblende and olivine pyroxenite termed the "Courtland" series. The emery occurs in a region in which mica-schist inclusions are abundant, and is in sharply defined veins immediately associated with rocks containing sillimanite, cordierite, garnet and quartz. A characteristic of these schist inclusions is the great abundance of biotite found around the ore in many places. The two chief mines are the Dalton and McCoy. They are worked by open-cut methods, some pits being over 80 ft. deep. In recent years production has been maintained by two operators, the Smith & Ellis Co. and the Keystone Emery Mills, but the former was the only producer of American emery in 1935 and 1936.

### *World*

*Greece.*—The island of Naxos is principally made up of gneisses and mica schists alternating with crystalline limestones. The lenticular masses of emery, varying in width from 15 to 150 ft. and sometimes as much as 100 yd. long, are closely associated with the limestone. The best Naxos emery is dark gray, usually mottled with bluish specks or streaks of pure corundum, and is the hardest emery known. The best ore comes from Vothrie, 9 miles from the coast, on the northeast side of the island. Another important deposit is at Apiranthos, 7 miles inland, from which the ore is shipped at the ports of Sulinos and Mutzoma. On the northern part of the island it is mined near Yasso.

Emery of a similar mottled appearance and quality, but of lamellated structure, occurs on the island of Nicaria, and a dark blue emery is found on the island of Samos, near the Asia Minor coast, but these deposits are not so extensive as those of Naxos. The mineral is sometimes also found embedded in white marble.

*Turkey.*—Emery is obtained in Asia Minor from the province of Aidin, which embraces almost the entire basins of the Sarabat and Mender Rivers. Smyrna is the principal port and center of trade for the surrounding districts and islands. Railways extend from the city into the interior along the basins of the rivers named above.

The greater part of the region is underlain by pure white or finely granular crystalline limestones, which are interfoliated with chloritic and micaceous schists and gneisses. The emery deposits occur as pockets or lenticular masses in the limestones and vary from a few feet to 200 ft. in width, and up to 300 ft. in length. The mineral also occurs as a detritus resulting from the weathering of the rocks.

Extensive deposits have been worked on the Gumuch-Dagh mountain in the vicinity of Ephesus and from the slopes of Ak Sivri, which is about 100 miles southeast of Smyrna. The emery from the former locality is usually fine grained, dark blue to purplish in color, and the interior of the masses is free from micaceous inclusions, while that from the latter

deposits is coarse grained and much darker in color. Kayabachi is at present the center of a number of small producing mines, the ores from which are shipped from the port of Kulluk. Other centers are Thyra, Aphrodisia, Inai and Azizieh, the shipping point being Smyrna. The ore exported is branded according to these seaports.

Usually the emery ore possesses numerous fractures or cleavage planes, so that it is easily broken into blocks of suitable size for handling. The main source of supply has been the loose emery or boulders embedded in a compact red clay, which occurs in shallow depressions in the limestone.

At the beginning of the war, the Abbotts' Emery Mines Ltd., the largest operator, installed a wire-rope tramway system from its principal mines to a more accessible point near the coast, but the plant was seized and destroyed by the Turks. All of the nine or ten producing mines were closed during the war, but in recent years production has been substantial.

*Russia.*—Four deposits of emery, some of which are being worked, occur in the southern portion of the middle Urals northeast of the Kyshtym works, on the eastern shores of Irtyash Lake, at the southern end of which the Techensky crushing mill is situated. All the emery deposits are confined to bands of crystalline limestones in contact with granitic gneiss, and occur therein as lenses or bodies ranging from 15 to 100 ft. in length, with a maximum depth of 150 ft. below the surface.<sup>11</sup>

*Other Countries.*—A small output of emery has been maintained annually from Wildenreuth, Bavaria. Emery occurs in several localities in Australia, mainly near Crookwell, New South Wales; also in the West Kimberley district of Western Australia. A fine-grained emery occurs in the Department of Minas, Uruguay, and also near Deschnet, eastern Fars, Persia, but no production from these localities has been reported.

#### POLITICAL AND COMMERCIAL CONTROL

The small output of emery in the United States is due mainly to the somewhat inferior quality of the known deposits as compared with those in Greece and Turkey and also to the low cost of mining in the latter countries. The Government of Greece controls the sale of emery from that country, which is effected by tender, all supplies being collected in its warehouse on the island of Syra. One of the largest exporters of both Greek and Turkish emery is at Amsterdam, Holland, where the crude ores are crushed and graded. The crude ore is also sent to five crushing and grading plants in the United States and some in England. There are no crushing plants in Turkey and only one small one in Greece.

#### PRODUCTION AND CONSUMPTION

##### *United States*

Emery has been produced in the United States since the early eighties, the first output being from Chester, Hampden County, Mass. The



Whittles, Va., deposit was worked between 1917 and 1928. The Peekskill deposits, Westchester County, New York, have been operated since 1883 and at present (1936) production is being maintained by one company in this district. During the World War annual production in the United States was about 15,000 tons, but has since gradually dwindled and in recent years the output has been under 200 tons, the lowest on record, but increased slightly to 325 tons in 1936.

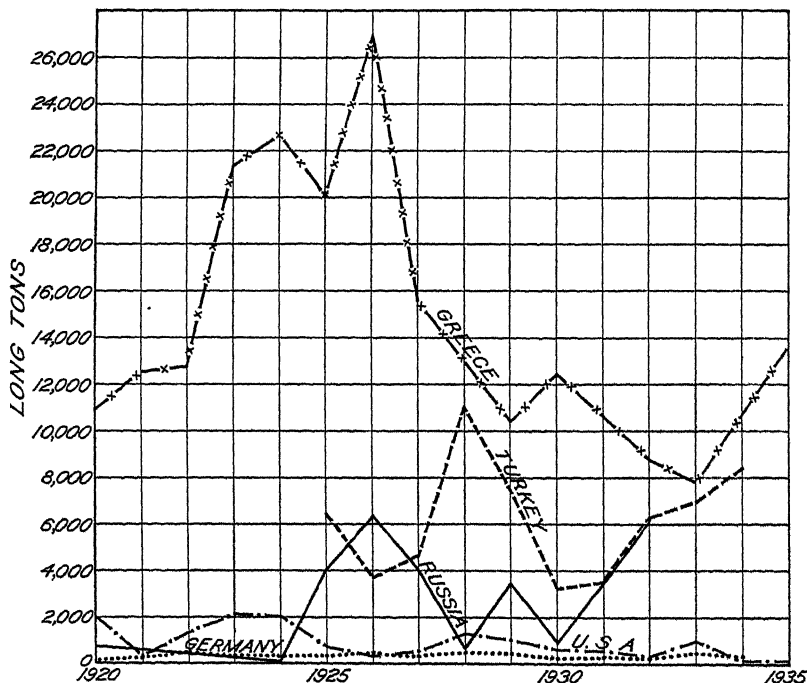


FIG. 1.—WORLD PRODUCTION OF EMERY, 1920-1935.

#### *World*

Emery has been mined in Greece for several centuries, but was not extensively produced until 1870. The prewar production ranged from 8,000 to 13,000 tons annually, reaching its maximum of nearly 27,000 long tons in 1926, and after an appreciable decline it has now risen to about one-half the maximum. The Turkish production has in the past been considerably less than that of Greece, but has remained comparatively steady and is now not far behind. A chart of the world's emery production is given in Fig. 1.

#### MINING METHODS

In the United States ordinary open-cast and underground methods of mining are employed in the Peekskill district. In Greece, mining operations are of the simplest character. The boulders or blocks containing

the ore, when not too large, are transported in their natural condition to the seacoast. The larger blocks are broken to a suitable size by means of hammers, sometimes with the assistance of heat and sudden cooling with water. Most of the present supply comes from the Liona and Mutzoma mines.

#### PREPARATION FOR MARKET

All the Greek and Turkish emery is shipped in lumps ranging from the size of a marble to 25 lb. in weight.

In the United States there are five emery-crushing and grading mills in which the crude ore, both American and imported, is first crushed by jaw crushers and thence through a series of chilled rolls to  $\frac{7}{8}$ ,  $\frac{3}{8}$  and  $\frac{3}{16}$ -in., then to finer sizes in ball mills. The mica is removed by allowing the coarse grain to fall down a chute; an uprushing current of air blows the mica to one side into a separate compartment. The emery then passes over a series of long troughs, shaking screens, whereby the emery is carefully graded into about 30 different sizes. The oversizes are further reduced by means of small rolls, and return through the circuit.

In some plants the crude grain is washed with water in pans of the Chilean mill type, in which hardwood wheels revolve. This cleans off the adhering dirt, and the cleaned material is dried and graded. Although this washing process is more costly, a much purer and better cutting article is produced. The overflow from the washing pans, containing all the fines, flows through a series of long settling troughs. The settled material is then shoveled out into drying ovens, and this "flour" is further screened into three or four grades. The finest flour-grades are made by drawing off the solutions at certain intervals of time, and drying the residue after final settlement. The 12 sizes from 6 to 46 mesh are termed "coarse grained" and the 12 sizes from 54 to 220, "fine grained" and there are four "flour" grades, from F to FFFF. There are also still finer flours, produced by decantation methods, which are used principally for optical work.

In the manufacture of emery paper or cloth, the largest size is No. 4 or 20 mesh and the following grading has been adopted:

Grade.....	4	$3\frac{1}{2}$	3	$2\frac{1}{2}$	2	$1\frac{1}{2}$	1	$\frac{1}{2}$	0	2/0	3/0	4/0
Mesh.....	20	24	36	46	60	70	80	90	120	150	180	220

The finer grades of paper keep their designation of multiples of F. In recent years acid treatment and roasting of the grain has considerably improved its abrasive qualities.

#### MARKETING, USES, SPECIFICATIONS AND PRICES

*Marketing.*—The sale of Greek or Naxos emery is under government control and buying is usually effected through agents on a commission

basis, which includes the making of all shipping arrangements. The buyer pays the costs of weighing and lighterage, which average 4s 6d per ton. Exports of Naxos in 1936 were mainly to the Netherlands, although there was a fair tonnage to France, Italy and the United States.

The imports of Greek and Turkish emery into the United States from 1930 to 1936 are as follows: Greece 1500, 1137, 2, nil, 742, 1455 and 650 tons and from Turkey 2300, 966, 600, 262, 2319, 2835 and 4891 tons.

For many years there has been more demand for Turkish than for Naxos emery, except in 1931 when the introduction of the acid-treated and heat-treated Naxos grain stimulated sales of the latter.

*Uses.*—Naxos emery does not undergo detrimental physical and chemical changes under intense heat, and the grains are very hard and sharp, therefore this emery is the most suitable for the manufacture of grinding wheels. The Turkish emery is usually slightly softer than Naxos and the grains tend to break down under pressure. As it is fairly tough, it is best suited for the manufacture of emery cloth or paper, in the setting up of polishing wheels and in pastes and compounds. In finer sizes the loose grains, both Naxos and Turkish, are used in grinding and beveling of glass. There are, however, some deposits in Turkey that contain an emery said to be harder than Naxos and to be usable for manufacture of grinding wheels. The American emery, being appreciably softer than either is now used mainly in pastes and compositions.

*Specifications.*—The specifications for an emery depend upon the uses to which it is to be put. Pure corundum or manufactured abrasives are considered too hard or harsh for certain grinding and polishing operations and it is the softer constituents of emery that tone down the harshness of the admixed corundum and give the desired results. The emery should contain corundum, those having the higher proportion being best suited for grinding wheels and those with the lower proportion for polishing and for pastes. The emery for the former should be hard and tough, whereas for the latter a more friable emery gives a smoother finish.

*Prices.*—Quotations in the mineral markets for the past few years have been steady: American crude ore, first grade, \$10 f.o.b. New York or \$16 delivered to grinders; Turkish and Naxos, \$30 to \$40 per ton in 350-lb. kegs delivered to grinders. American grain  $4\frac{1}{2}\text{¢}$  and foreign grain 6 to 7¢ per pound f.o.b. grinders.

The recent United States import values were \$12 to \$13 per ton for Turkish and \$24 to \$25.40 for Naxos. The 1936 selling rates of Naxos were: first quality, large lumps, £4 8s; small lumps, £4 14s; second quality, large lumps £4 6s; small, £4; emery fragments (chips), £3 13s per metric ton. These prices apply to deliveries ex-warehouse at Syra.

#### GARNET

The name "garnet" is given to a certain group of minerals possessing similar physical properties and crystal forms, though their chemical

compositions vary widely. The term is derived from the latin word *granatus*, meaning seedlike, because the crystals embedded in the matrix usually resemble seeds. Before the eighteenth century, the mineral was known as "granat."

### COMPOSITION

The garnet group comprises seven different species, all of which are silicates of aluminum, calcium, iron, magnesium, manganese or chromium, the different metals being replaceable one by the other. Because of the variety in their compositions, some garnets are unaltered by weathering while others become completely disintegrated. Very often other minerals, particularly quartz, mica and pyroxene, are included within the garnet crystals, and in some instances these are so finely disseminated that it is almost impossible to make a mechanical separation of the garnet. Varieties of garnet are as follows:

Almandite ( $3\text{FeO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$ ) the commonest and andradite ( $3\text{CaO} \cdot \text{Fe}_2\text{O}_3 \cdot 3\text{SiO}_2$ ), also quite common, range in color from deep red, brown to black. The colored varieties of andradite are melanite (black), demantoid (green), topazolite (yellow-green). Grossularite ( $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$ ) may be white, pale green or yellow. Uvarovite ( $3\text{CaO} \cdot \text{Cr}_2\text{O}_3 \cdot 3\text{SiO}_2$ ) is emerald green. Pyrope ( $3\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) ranges in color from deep red to black. Spessartite ( $3\text{MnO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$ ) is brown to red in color. Rhodolite, a mixture of two molecules of pyrope to one of almandite, varies from pink to dark red.

### PROPERTIES

*Color.*—As outlined above under different varieties.

*Hardness.*—Hardness varies from 6 (grossularite) to 7.5 (almandite, uvarovite and rhodolite), though some varieties of almandite are said to be 8.

*Crystal System.*—Cubic, commonly as rhombic dodecahedrons or tetragonal trisoctahedrons, or in combination of the two.

*Cleavage.*—Occasionally an indistinct dodecahedral cleavage is observed, but some species of almandite possess a pronounced laminated structure, which forms planes of weakness along which the mineral separates, but this parting has no relation to the crystal form and is not a true cleavage.<sup>20</sup>

*Fracture.*—Garnets possessing a glassy structure usually have a marked conchoidal fracture but sometimes the mineral tends to break into thin flakes (which are probably parting planes), while in other varieties the fracture is sharp and uneven.

*Index of Refraction* in grossularite is 1.735; almandite 1.778 to 1.830; andradite 1.865 to 1.94.

*Fusibility.*—High-iron garnets readily fuse to a dark glass—almandite at 1315° C.—while high-chromium garnets are unfusible before the blowpipe.

*Tenacity.*—Aggregates of crystals, or the laminated varieties, fracture readily into their component parts, but the solid and well formed crystals are very tough.

*Other Properties.*—Specific gravity, 3.5 to 4.2. Luster, vitreous resinous or dull. Transparent to opaque. Molecular weight: pyrope, 405; almandite, 499; andradite, 509. Streak, white. Dispersion, 0.24 to 0.28.

### ORIGIN AND MODE OF OCCURRENCE

Garnets occur as accessory minerals in a large variety of rocks all over the world, but are particularly common in gneisses and schists. In some instances they are present in such large quantities that the rocks are designated as garnet schists or gneisses. They also occur as contact-metamorphic deposits in crystalline limestones, pegmatites and serpentines, etc. The usual associated minerals are hornblende, mica and quartz, with feldspar and pyroxenes in smaller proportions. Little information is available as to the occurrence of garnet below 400 ft. in depth, because nearly all garnet mining has been confined to open-cut methods, although garnet is sometimes found in the gangue at moderate depths in mining other ores, such as iron and copper. Garnets occurring in contact-metamorphic zones, which are formed by the replacement of the sedimentary rocks, are of secondary origin, but it is probable that the garnet in pegmatites and granites is one of the original rock minerals and of primary origin.

Because they are more resistant to weathering and erosion than their associated rocks, the garnets often occur as a detritus of crystals in the immediate vicinity of the original rocks, or as rounded grains in river and sea sands. In sands, owing to their comparatively high specific gravity, the garnets are usually concentrated by water action into beds or layers.

Garnets vary greatly both in size and in concentration. The crystals may be several feet in diameter—like those being mined in New York State—down to crystals smaller than a pin's head. They may occur very sparsely distributed or close enough to constitute 70 per cent of the rock. In some places there is high concentration of very small crystals as pockets or lenses and in others the garnet is in the massive form that does not disintegrate or break up into individual crystals.

### DISTRIBUTION OF DEPOSITS

Garnet in its various types is not an uncommon constituent of the rocks throughout the world, but deposits of high-quality abrasive are comparatively rare. The world's commercial abrasive garnet deposits

appear to be confined mainly to the American continent. Although there are numerous occurrences of possible abrasive garnet in other countries, little has been done towards their development, and there has been no recorded production outside America, except from Spain and small quantities from India and Madagascar. For commercial occurrences of gem garnet, see chapter XVI.

### *United States*

The supply of commercial abrasive garnet at present (1936) comes from two deposits in New York State operated by the Barton Mines Corporation and by the Warren County Garnet Mills, Inc., and from one deposit in New Hampshire operated by the Davenport Garnet Co. Between 1922 and 1925, when production was at its peak, there were several other operators in these states as well as one in North Carolina.

*New York, New Hampshire and North Carolina.*—New York State is the principal producer, the high-quality material of the Adirondack district being considered the world standard for abrasive garnet. Garnetiferous rocks occur over a wide area in Warren, Essex and Hamilton Counties. At the Barton mine the mineral is in fairly well developed crystals from 1 in. to 3 ft. in diameter, and occasionally in large masses almost devoid of individual crystallization. The rocks are mainly metamorphosed gneisses and the garnet content seldom above 15 per cent, except in deposits in which massive aggregates are present. In New Hampshire, the garnet as crystals the size of a pea or slightly larger constitutes an average of 50 per cent of the porphyritic biotite schists in which they occur.

The following brief descriptions of the New York, New Hampshire and North Carolina deposits that were being operated in 1925 are here-with quoted from Myers and Anderson's report.<sup>20</sup> These descriptions give the locations, geology, characteristics of the garnet and some methods of mining and handling the ore.

*The Barton Mines Corporation.*—The quarry of the Barton Mines Corporation is on Gore Mountain, about 11 miles by road from the village of North Creek. The most characteristic feature of the dark gray, massive rock quarried is the garnet crystals themselves, which give the rock a porphyritic texture. Mineralization is simple, as hornblende constitutes nearly 40 per cent of the rock mass, the remainder being divided between orthoclase and plagioclase feldspars, pyroxene and mica. Small amounts of pyrite and magnetite are also present. The garnet content of the ore averages about 13 per cent and occurs in crystals of unusual size. Single crystals a foot in diameter are common and individual ones from 30 to 36 in. in diameter have been found. The crystals have a very pronounced laminated structure by which they are divided into plates from a sixteenth to a quarter of an inch in thickness.

The garnetiferous rock has undergone considerable alteration at the surface, and the first mining was in this oxidized zone from which the garnet could be readily recovered. Later quarrying was started in the unoxidized rock, and with the erection of a modern concentrating mill in 1924, the quarrying was placed on a systematic basis. The quarry

now consists of a series of open pits which are being developed into regular benches. Ten-foot holes are drilled with jackhammers and these are shot with 40 and 60 per cent gelatin. Boulders are blockholed with small charges of dynamite. The broken ore is loaded in three-ton cars with steam shovels and hauled to the mill with a gasoline locomotive. In breaking the ore in the quarry many of the garnet crystals are shattered. The largest fragments of clean garnet are picked and sacked for immediate shipment.

A new lower level in the mine has now developed ore at a depth of 380 ft. below the surface. For further details see the article by T. S. Mennie.<sup>18</sup>

*The North River Garnet Co.*—The quarry and mills of the North River Garnet Co. are located on the eastern side of Thirteenth lake in Warren County about 10 miles from North Creek, the nearest shipping point. The gneiss quarried carries 4 to 8 per cent garnet in crystals having a maximum diameter of three inches. Hornblende and feldspar constitute the most important gangue minerals. The quarry is in a large projecting knob of gneiss, and has a maximum diameter approaching 300 ft. The jointed structure of the rock is highly developed, and the horizontal partings are utilized as the base of small benches in quarrying. The rock is drilled to a depth of 15 ft. and holes are sprung and then loaded with 60 per cent ammonia dynamite which is fired with a blasting machine. Large blocks are reduced by blockholing and the broken ore is loaded either by hand or by steam shovels to quarry cars which run to the head of the mill, a maximum distance of 200 yards.

For further details see article by F. E. Wormser.<sup>23</sup> The North River Garnet Co. was amalgamated in 1927 with the Barton Mines Corporation, when mining and milling operations were restricted to the latter property. For a number of years the Barton Mines Corporation has continued to be the principal producer of garnet.

*Warren County Garnet Mills.*—The Warren County Garnet Mills, Inc., operate a number of small scattered quarries in the vicinity of Weverton and Johnsburg, 6 miles southeast of Gore mountain. The garnet occurs in broad bands in a biotite gneiss, much of it being in massive aggregates which in places constitute nearly the whole rock. The garnet ore is removed from shallow excavations, seldom exceeding 8 feet in depth. The rock is drilled by hand, with a single-jack, blasted, sledged and hand-picked, and trucked to the mill. The garnet content of the ore going to the mill is high, varying from 30 to 60 per cent.

This property has been operating for a number of years, maintaining a small but steady output.

*The Wausau Abrasive Co.* (Davenport Garnet Co.).—The mine and mill of the Wausau Abrasive Co. is located in North Wilmot, Merrimack County. The garnet is present as numerous small crystals,  $\frac{1}{4}$  to  $\frac{3}{8}$  in. in diameter, forming 40 to 60 per cent of the total rock mass. Feldspar and biotite mica are the most prominent gangue minerals. The quarry is approximately 180 by 100 ft. with a maximum depth of 25 ft. Holes are drilled to a depth of 6 ft. and shot with 60 and 75 per cent gelatin. Boulders are bulldozed, and the broken rock is loaded by hand into cars, which are hoisted up an incline. The cars are then trammed by hand to an ore bin on the hillside above the mill. The ore from the bin is fed to a jaw crusher which crushes to  $1\frac{1}{4}$  in. The crushed ore falls into a loading bin which loads the buckets of an aerial tramway. This tramway conveys the ore to the mill, a distance of 1200 feet.

After the reorganization in 1929, the company acquired the name of

Garnet Products Co., a mining subsidiary of the Wausau Abrasive (sand-paper) Co., but since 1935 has been known as the Davenport Garnet Co. For full details of the New Hampshire garnet deposits, see the recent article by L. C. Conant.<sup>14</sup>

The deposit of rhodolite garnet at Sugar Loaf Mountain,  $2\frac{1}{2}$  miles south of Willets, N. C., has been worked at intervals by the Rhodolite Company, and a considerable tonnage of garnet was recovered between 1900 and 1926. The mineral, in small crystals of less than  $\frac{1}{2}$ -in. diameter, occurs in a large body of quartz feldspar mica schist. The average content is 20 to 25 per cent garnet with local concentrations as high as 60 per cent. A mill was erected in 1925 but for the past 10 years there has been no production.

*Other States.*—The following is a brief account of the garnet activities in other states during the past five years, but no sales have been recorded. Prospecting work was done in 1930–1931 by the Pacific Coast Garnet Co. on an amber colored andradite garnet deposit northwest of Cadiz station, San Bernardino County, California. For the past three years J. I. Fassett has been prospecting a garnet property at the Sunny Day mine, near Indio, Riverside County, California, but although a few tons of rock have been mined, there have been no shipments. The Vermont Mica Co. during 1931–1932 operated a micaceous gneiss carrying about 14 per cent garnet at Gassets, Vt. The company marketed the mica and stored the garnet for future sales. In 1936, the Celo Mines Inc., Burnsville, N. C., produced a small amount of garnet as a by-product in the concentration of kyanite rock. The pre-Cambrian schists of North Georgia constitute a possible source of abrasive garnet and are said to be worthy of investigation.

In Alaska, at Rothsay point, which is at the entrance to the Stikine River opposite Wrangell, beautifully formed, almost transparent claret colored crystals,  $\frac{1}{2}$  to 1 in. in diameter, occur in a fine-grained dark gray mica schist. The garnets are studded throughout the schist at fairly close and regular intervals and readily fall out when the rock is fractured. The garnets possess excellent abrasive qualities and about 10 to 15 years ago small shipments were made to various places on the west coast.

### *World*

Garnet occurs in many countries throughout the world, but during the past decade production of garnet outside the United States has been comparatively insignificant. In recent years a small output has been maintained from Spain and India (garnet sands).

*Canada.*—Garnetiferous rocks, mainly gneisses, occur in many parts of Canada. In some localities, particularly in southern Quebec, garnets are a common rock constituent covering many square miles of territory. Prospecting has been carried out intermittently on several deposits, but the only output of consequence has been from a deposit in Ashby Town-



ship, Lennox and Addington Counties, Ontario, when in 1923-1924 about 1200 tons of rough concentrate was shipped. A deep red garnet of exceptionally high quality occurs intimately associated with pyrrhotite near Labelle, 100 miles north of Montreal, Quebec. Prospecting has been carried out intermittently during the past 10 years but only small trial shipments have been made. The ore occurs in a garnetiferous gneiss. The garnets of the latter are an inferior abrasive and are difficult to separate from the high-quality mineral except by hand-picking. A few car lots of garnet rock obtained from a neighboring deposit near Labelle were crushed in a mill erected on the property during 1934 and shipped for sandblasting purposes. Prospecting work has also been conducted on a garnet deposit at Depot Harbour on Parry Island, Ontario; near Sudbury, Ont. and at Cheggogin Point, Yarmouth, Nova Scotia. Full details of all the known Canadian garnet occurrences (about 100) will be found in the Mines Branch Bulletin.<sup>13</sup>

*Spain.*—The Spanish garnets, which are a pale pink, occur as small rounded crystals in the alluvial deposits of the Province of Almeria. The garnets are inferior to the American and when crushed do not yield the full range of the sizes required by users. At one time imports of these garnets to the United States amounted to 500 to 3000 tons annually, but have steadily dwindled in the past decade, and ceased altogether since 1933.

*India.*—Garnetiferous gneisses and schists occur in many parts of India. Many years ago an attempt was made to market a massive garnet rock in the Hazarbagh district of Behar, but it did not prove remunerative. No regular garnet industry has been established and the supply now comes from local concentrations of river and sea sands. The principal places at which production has taken place are in the Tinnevely district of Madras, where over 1000 tons of garnet sand was produced in 1914; Nellore in the Deccan, and a number of different localities in Mysore. For the past few years there has been a fairly steady annual production of between 200 and 300 tons of garnet sand from the Tinnevely district.<sup>16</sup> There are numerous deposits of gem and ornamental garnets in India and production has been fairly steady.

*Other Countries.*—Small and irregular shipments have been made from Madagascar. Tests have been conducted on garnet from deposits in Ceylon, South Africa, Malawe Hill in Nyassaland, British Somaliland, from an island in St. Huberts Bay off the Labrador coast, etc. Near Prague, Czechoslovakia, alluvial garnet (pyrope) has been washed and concentrated in crude jigs. Mittleburge, Czechoslovakia, was at one time the world center for gem garnet, 10,000 men being employed.

#### POLITICAL AND COMMERCIAL CONTROL

The abrasive garnet industry, which reached its peak in 1923-1924, has dwindled regularly and appreciably, owing largely to the competition

from manufactured abrasives. The comparatively small output between 1931 and 1935, however, increased about 75 per cent during that period; while the output of the competitive manufactured products, silicon carbide and fused-alumina abrasives, increased 144 per cent for the same period. The United States is now the only producer of mined garnet that is crushed and graded, and the product is recognized as the world standard. Imports of garnet into the United States, which at one time were quite substantial,

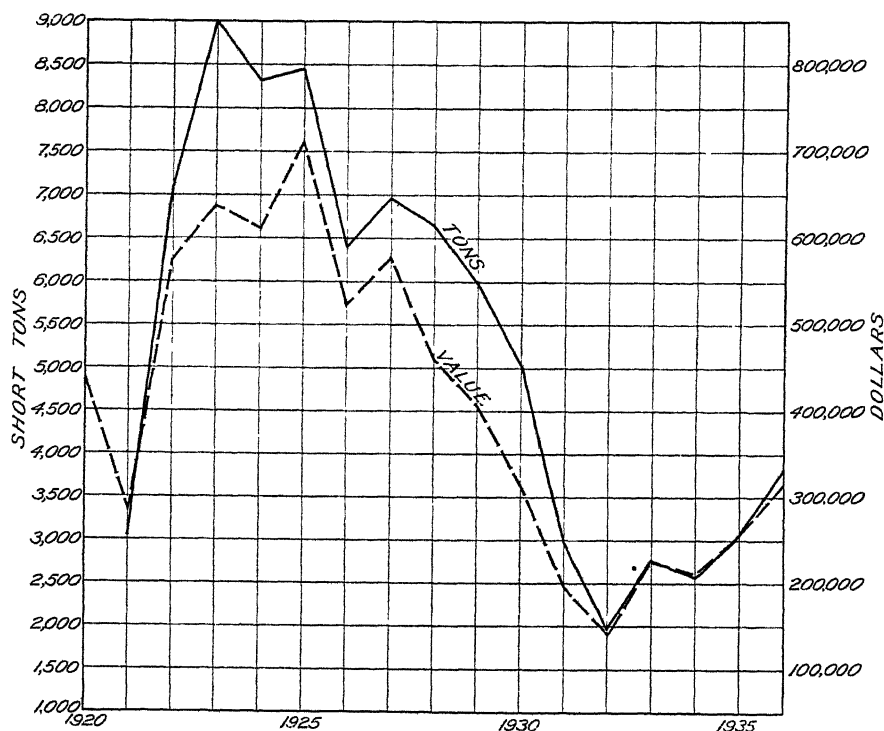


FIG. 2.—UNITED STATES PRODUCTION OF GARNET, 1920-1936.

have now ceased entirely. The available supply and the capacity of existing mills are far greater than the present demand. The principal sandpaper manufacturers in the United States own or control the operating mines.

Crude garnet ore and ungraded concentrate enter the United States on the free list under Paragraph 1619 of the Tariff Act. Graded and specially prepared garnet is dutiable at 1¢ per pound under Paragraph 1415.

#### PRODUCTION AND CONSUMPTION

*United States.*—Garnet was first employed as an abrasive in the United States and became commercially important about 1880, when it was used

as coated abrasive by Herman Behr and Co., of Brooklyn, N. Y., and its superiority over flint and the California red carnelian for sanding hardwood was quickly established. The first mining operations were conducted by H. H. Barton, of Philadelphia (to whom the initial developments of the abrasive garnet industry are largely due), in the Adirondack section of New York State, followed immediately afterwards by the development of a deposit by Herman Behr at Boothwyn, Delaware County, Pennsylvania, and later, of deposits in Connecticut. The first large-scale milling of garnet ores was done by F. C. Hooper, of the North River Garnet Co., North River, N. Y., in 1893. After the exhaustion of the deposits in Pennsylvania and Connecticut, production was confined to New York, New Hampshire and North Carolina. The New York mines are now the world's leading producers.

United States production of abrasive garnet did not attain commercial importance until after the milling operations in 1893 referred to above. The total garnet production from 1893 to 1936 amounted to 192,889 short tons.

Fig. 2 gives the output of the United States from 1920 to date, and illustrates the decline since the peak year of 1923.

*Other Countries.*—The production of abrasive garnet in countries outside the United States has been small and intermittent. The following is the record in long tons since 1920:

Year	Long Tons				
	Canada	India (Garnet Sand)	Spain	Japan (Garnet Sand)	Madagascar
1920		20	195		
1921		5	5		
1922				3,351	10
1923	1,116	3	966	3,529	5
1924	321		1,094	3,474	
1925				3,484	
1926		14			
1928		480			
1930		7			
1932		147			
1933		295			
1934		225			
1935			179		

#### PROSPECTING, EXPLORATION, AND MINING METHODS

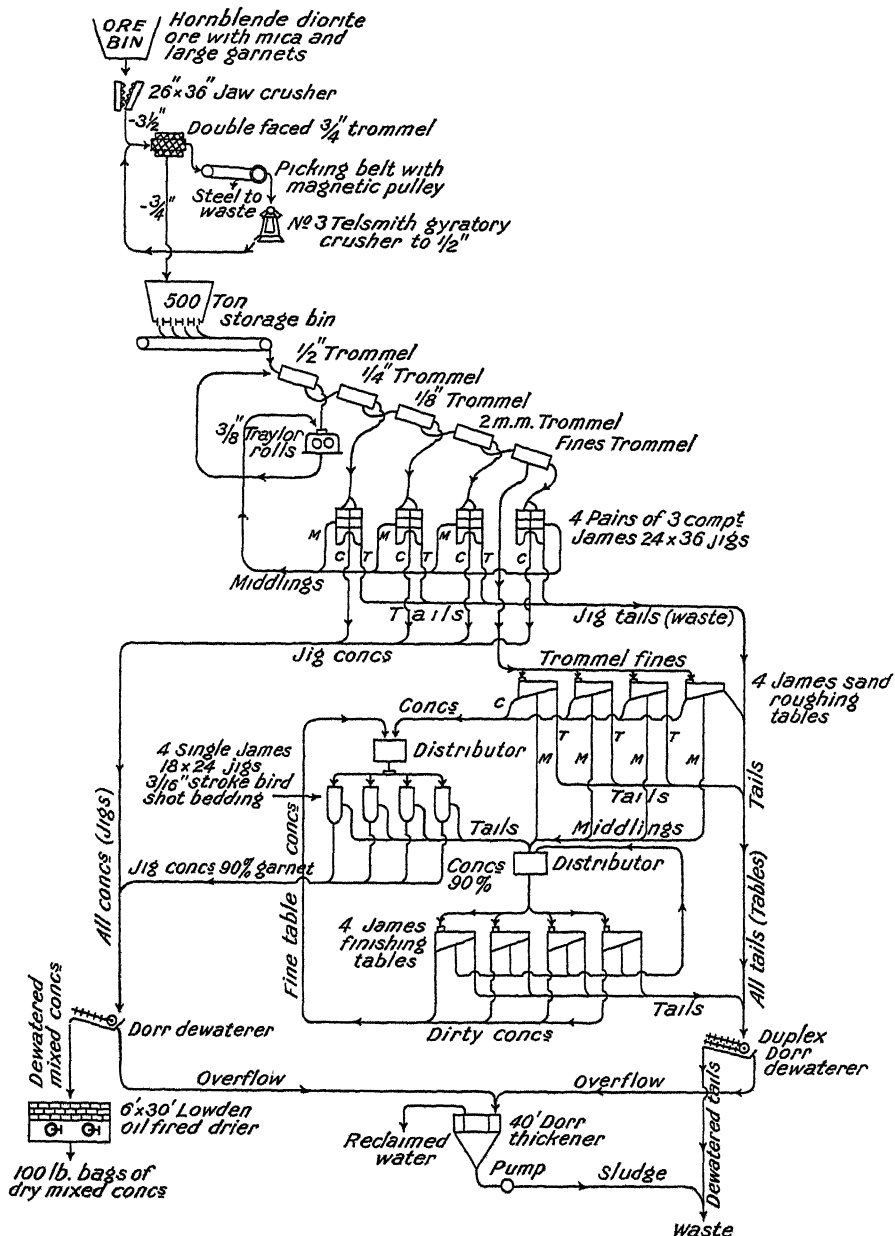
Because present demand for abrasive garnet is comparatively small and is greatly exceeded by the known potential supply, special search for new deposits would not appear to be necessary. No deposits need be

considered unless preliminary examination indicates that the garnet will adequately fulfill the qualifications as outlined later in this chapter. If the surface indications are satisfactory, samples for tests should be taken, if possible, at a depth below the influence of weathering.

Garnet is usually mined by open-quarry methods, though in some instances it has been extracted by underground stoping. The usual procedure is followed, though in deposits containing very large crystals excessive shattering through the use of too much powder should be avoided and hand-picking is advantageous. If the garnet occurs as concentrated bands or zones, the deposit should be cross-trenched and the best zones followed. The mining practices of some of the producers in the United States is briefly outlined under the descriptions of the individual properties.

#### PREPARATION FOR MARKET

Before being used for the abrasive coating of papers and cloths, the garnet must be concentrated and graded. Being a heavy mineral (av. sp. gr. 4.0), garnet is usually concentrated by ordinary gravity methods, either wet or dry. The former is the general practice and the more suitable for the majority of the ores. The main object in concentrating garnet for the paper trade is to obtain a material with as large a mesh and as pure as possible, and to avoid fines. The manufacturers of coated abrasives in the United States usually prefer to do their own grading (sizing), so they require coarse material from which varying proportions of any size can be obtained by crushing, but in crushing, fines are unavoidably produced. When standard grading is more general, these grades may possibly be made direct in the course of concentration, but the present practice in milling is to mix together and bag all the grades obtained. The degree and stages of crushing are of the utmost importance, therefore, and care must be taken that the coarse particles of garnet are free from attached impurities. Therefore it is necessary that preliminary concentration tests be conducted on a garnet ore in order to determine the most efficient degree and type of crushing before the erection of the mill. A flowsheet diagram of the leading garnet producer of the United States in 1925 is given in Fig. 3. Some changes and improvements have been made since then, but these illustrate the careful crushing and screening, followed (in the wet process) by jigs and table concentration, that are necessary to preserve the large garnet grain and reduce fines to the minimum. Magnetic separators are used in the dry stages to extract the magnetite, pyrrhotite and included iron. The dry process differs essentially from the wet in the use of an air concentration table of the Sutton, Steele & Steele type. A preliminary extraction of the mica is accomplished by allowing a stream of the dry crushed ore to encounter a crosscurrent of air that deflects the lighter particles of mica and excess fines.



*Ore, Large laminated and masses of garnet crystals which average 12% of the rock composed principally of hornblende with some feldspar, pyroxene and mica*

FIG. 3.—FLOWSHEET OF 250-TON GARNET MILL, THE BARTON MINES CORPORATION, NORTH CREEK, WARREN COUNTY, N. Y., IN 1925.

*Impurities.*—Quartz, feldspar and mica, being considerably lighter than garnet, should be entirely removed during the process of gravity treatment. Iron pyrites and pyrrhotite can be removed by careful tabling and the latter by ordinary magnetic separation. Hornblende is one of the commonest impurities and, as its specific gravity (3.4) is close to that of garnet, it is rarely, if ever, entirely eliminated from the final concentrates. It can, however, be effectively removed by means of the Ulrich magnetic separator.<sup>13</sup> Hornblende, though softer than garnet, is nevertheless fairly hard and in small quantities is not detrimental. The black grains almost invariably present in the commercial red or pink garnet-coated papers and cloths are hornblende.

Details of garnet flowsheets, machinery and concentration tests will be found in various reports.<sup>13, 17, 18, 20</sup>

*Heat-treatment.*—The toughness, fracture and color of iron garnets are improved by heating the grain at about 800° C. for about 12 hr. and then quenching. This treatment, covered by U. S. Pat. 1836448, Sept. 22, 1930, and by U. S. Pat. 2029253, Aug. 30, 1935, is successfully employed by several manufacturers of abrasive-coated papers and cloths.

*Grading.*—Grading consists of alternately crushing and screening the concentrate in order to obtain the full range of grades (mesh sizes) required by the sandpaper manufacturers.

#### TESTS AND SPECIFICATIONS

*Qualifications of Abrasive Garnet.*—Garnets vary considerably as to color, hardness, toughness and method of fracture. For the best abrasive, it should be as hard as possible, at least 7.5 (quartz is 7.0). When the garnet is crushed the grains should break into sharp, angular fragments without curved or rounded edges. The mineral should not break too easily, but it should be brittle enough so that the individual grains will eventually break and form new cutting edges rather than become rounded under the strain of work. Thin or slivery grains do not arrange themselves correctly because they tend to lie too flat when applied to the glued surface of paper, thus presenting a smooth, noncutting surface, or they protrude above the surrounding grains and tend to scratch. The crushed garnet grains should have a high capillary attraction, so that the glue will completely cover and adhere to them when they are being fastened to the paper or cloth. The color does not seem to have any particular bearing on the abrasive qualities, but the deep red or wine colored mineral is always preferred. In order to obtain the full range of grades necessary for coated papers, the garnet should be capable of being broken into comparatively large (size of a pea), clean and solid pieces free from embedded impurities, and with the minimum amount of fines. Therefore deposits that contain garnets of small crystals are of little or no commercial value for sandpapers, no matter how great the garnet content of

the rock may be. Granular garnet usually breaks into rounded grains; other deposits containing large crystals that are badly shattered, or crystals containing embedded impurities, would with little pressure crumble almost to dust. The compact, massive garnet might be used commercially, but comparatively little experimental work has been done on that type. Massive garnet, however, is almost invariably off-color, and the toughness and hardness of the crushed grains are usually inferior to those of solid garnet crystals. The grains in garnet sands are too small and they are rounded by erosion and water action.

Briefly, therefore, the best abrasive garnet comes from deposits of clean, unfractured, individual deep red crystals at least the size of a pea, but preferably larger. The recovery from the average types of deposits should be at least 10 per cent of such garnet in order to be commercially valuable. Up to the present, the highest quality of abrasive material has been obtained from the large, laminated crystals or boulders of red garnet like those in New York State. The kinds used for abrasive purposes are all iron garnets. Almandite is the commonest, but andradite and rhodolite are also used.

*Tests for Abrasive Utility of Garnet.*—There is no efficient method of testing the abrasive quality of garnet or of any other loose-grain abrasive. The real test is in its practical application. There are, however, several rough tests and examinations that serve to indicate their abrasive possibilities. The size, fracture, shape and purity of the grains, as outlined above, can be determined or compared by the microscope. The *toughness* of different garnets may be roughly compared by reducing them to the same grade (No. 1) and drawing a knife blade over a small quantity placed on a piece of steel or glass. Soft garnets will drag and soon break up into powder, but the knife will ride over the tough particles without appreciably reducing their size.

*Capillarity.*—This test depends on the capillarity of crushed garnet and serves to indicate its tenacity when applied to the glued surfaces of the paper or cloth. The higher the capillarity, the tighter will each particle adhere when coated with glue. In making comparisons care should be taken that the grains are of the same mesh and are absolutely dry, clean, and free from dust. The apparatus consists simply of a glass tube about 10 in. long, 4 or 5 mm. inside diameter, blocked at one end by a piece of fine screen. The tube, which should be dry and clean, is partly filled with the sample to be tested, gently shaken down, and the closed end is dipped into a known height of water (about  $\frac{1}{2}$  in.). The water will rise up through the screen and into the garnet, and after 3 to 4 minutes the tube may be removed from the water and the dry garnet poured out. The remainder, which adheres to the inside of the tube, is measured. The height of water into which it was placed should be deducted.

A capillarity scale of the different sizes is given in the Mines Branch, Canada, report.<sup>13</sup> Briefly, the rise in centimeters for the coarse sizes is about 4.0 cm.; for the medium, about 8.0 cm., and for the fines 13.0 cm.

*Testing Machines.*—After a garnet has passed the above preliminary test, it may be tried out by applying the graded grain to a belt or disk. The glue used and the method of adhering the garnet to the backing is almost as important as the quality of the abrasive itself, so that comparisons should preferably be made only after the test samples have been made by a manufacturer of coated papers. The machine used by the Mines Branch, Ottawa, consists of a wooden drum 1 ft. in diameter and 1 ft. wide, around which the coated paper is stretched. A weighed block of hardwood to which are attached a lever and weight (to give the required constant pressure) is placed on the drum, which is made to revolve at a certain speed and time, the wooden test block moving gradually across the face of the drum during the operation. After each test the paper is examined for condition and the wooden block weighed to ascertain the loss. A strip of standard paper of the same grade (mesh) as the test strips is used for comparison. The methods and results of the testing of garnet for both sandpaper and sandblasting are given in various reports issued by the Ore Dressing Division, Department of Mines, Canada.

#### MARKETING, USES AND PRICES

Probably about 90 per cent of the abrasive garnet is used for the manufacture of abrasive-coated papers and cloths and the remainder as a lower priced material in the form of loose grain for various purposes, such as surfacing and polishing marble, slate, soapstone, and other soft stones; in some sandblast operations and a very small amount for the surfacing of plate glass.

*Papers and Cloths.*—On the American continent garnet-coated papers and cloths are used in the woodworking industries, particularly for hardwoods. In abrading softwoods, garnet and quartz papers appear to be nearly equally efficient, as both are soon clogged with the wood particles; consequently there is no advantage in the higher priced garnet. The recent introduction of open-coated papers, however, largely overcomes this trouble. In hardwoods the superiority of garnet over quartz is most pronounced and the cutting efficiency of the former is said to be from two to six times that of the latter. These papers are also used for the finishing of hard rubber and celluloid, for felt and silk hats, and as fine disks in dental work. They are employed extensively on leather, particularly in the boot and shoe industry for the scouring of heels and soles. In recent years fine grits of waterproof garnet papers have taken the place of pumice in the rubbing down of varnished and painted surfaces, especially automobile bodies. Garnet cloths are sometimes used for the softer metals.



The garnet-coated abrasive is used in the form of belts, covers for drums, disks, and as small sheets in hand work. The different grades of garnet used on these papers and cloths range from No. 5, the coarsest, which is about 15 mesh, to 7/0, which is about 220 mesh.

There are about ten manufacturers of abrasive-coated papers in the United States, two in Canada, and several in England, France, Germany and other European countries. Details of the manufacture of coated papers are given in part IV of the Mines Branch Abrasives report.<sup>25</sup> The recently introduced method of electrostatic coating is meeting with success. In this process an electrostatic machine causes the grain to jump up and embed itself in the wet, glued surface of the moving backing. It is claimed that the grains stand up on their edges and are more evenly surfaced, owing to individual grain repulsion. This is a distinct advantage over the old method of coating, particularly if flat or slivery grains are present.

*Loose Grain.*—A small amount of garnet grain is bonded into wheels made by low-temperature processes which are employed for grinding glass and some metals. Some sales are made annually within the United States and Canada for crushed and sized garnet ore for sandblasting stone monuments (inscriptions) and on certain metals. A good, tough garnet from very rich deposits that would not require the expense of concentration might prove an efficient competitor of silica sand or of the more expensive metallic or manufactured abrasives.

*Glass Surfacing.*—The excess fines produced in manufacturing abrasive coatings, as well as finely crushed and water-settled garnet ore, was utilized for some years in the United States for surfacing plate glass in the stage between the coarse sanding and the final rouge polishing. Garnet was introduced for this purpose in 1914 and between 1925 and 1934 a fair tonnage was consumed by the glass industry. The Ford Motor Co. was the largest user, and operated its own deposit at Danbury, N. H., solely for this purpose, but in 1935 the company discontinued this use of garnet and reverted to graded sand. It is believed that very little garnet is now being used for glass surfacing.

*Gem Garnet.*—Clear, flawless garnets of various colors are extensively used for ornamental purposes. Between 1883 and 1920, the value of gem garnet produced in the United States varied from \$2,000 to \$22,000 in a year, the high values being obtained from the rhodolite garnet in North Carolina during 1900 and 1901. The output in the United States has dwindled to a figure so unimportant that no records have been kept since 1921. A large quantity of garnet is used for the jewels of the cheaper watch movements, but none has been produced in America for this purpose except a small amount during the World War, because the foreign supply has been abundant and its prices lower than the cost of domestic

production. Further details are given in chapter XVI and also in U. S. Bureau of Mines *Bulletin* 256.<sup>20</sup>

*Markets.*—The demand for garnet (which is indicated under Uses) has dropped steadily and appreciably since the peak year of 1923, owing to competition from manufactured abrasives. A small increase since 1931 has been due mainly to general improvement in business and perhaps to a small extent to the improvement in garnet grain through the heat-treating process and electrostatic method of application.

In Europe garnet is not so extensively used as on the American continent. This may be attributed to the inferior quality of the Spanish garnet, to the high price of the American, and to the use of chalk flint, which is cheaper than garnet and occurs in great abundance in England and in France. It is superior to the American so-called "flint" (which is really quartz) and is almost equal to garnet in its abrasive qualities. The European manufacturers of coated abrasive require graded grain, whereas the American manufacturers buy concentrates from the garnet producers and grade it themselves. The Barton company recently installed a grading plant at its North River mill.

*Prices.*—The price of the best American garnet in the form of a high-grade mixed concentrate (over 90 per cent garnet), free of fines, has remained constant for the past decade at about \$85 and lower grades \$60 per ton in bags of 100 to 150 lb. f.o.b. shipping point; glass-surfacing fines, \$40 to \$45. The finished and graded grains vary from 4¢ to 8¢ per pound, depending on grade or size of grain. The inferior kinds of garnet, such as the Spanish and low-grade concentrate, sell for about \$50 per ton.

#### SILICEOUS OR OTHER NATURAL ABRASIVES

Silica in many forms is used for abrasive purposes; i.e., flint, quartz and quartzite, chert, sandstone, sand, silt, amorphous silica, tripoli, diatomite, rottenstone and siliceous clays. These materials depend mainly upon the free silica and sharpness of the grains for their abrasive properties.

The abrasive uses and the common forms of silica as well as some other natural rock and materials are briefly listed in the table shown on p. 34.

Grindstones and pulpstones are often classified together under one heading, particularly for import and export figures. Although both are made from sandstone they are used for entirely different purposes and deposits that furnish the one rarely, if ever, furnish the other. Therefore these materials, as well as sharpening stones, millstones, etc., are reviewed separately.

#### GRINDSTONES

Natural grindstones are made from certain sandstones that occur throughout the world. The proper selection of the sandstone is of the

greatest importance and depends on the use to which the finished grindstone is to be applied. The suitability of a sandstone for the grinding of different substances depends on: (1) the percentage, as well as upon the character of the cementing materials such as silica, lime, clay, iron oxides, etc., (2) the size and shape of the abrasive (quartz) grains.

ABRASIVE USES	TYPES OF SILICA USED
Grindstones, pulpstones and sharpening stones	Massive sandstone from very fine to moderately coarse-grained. Also novaculite, fine-grained schist and siliceous shales
Millstones, burrstones, chaser stones	Cellular quartzite, fine-grained quartzite, quartz conglomerate, granite, silicified limestone
Tube-mill grinding pebbles	Rounded flint, quartzite and granite pebbles, also artificially rounded quartzite blocks
Tube-mill lining	Chert, flint and quartzite in dense, solid blocks.
In sandblast work	Quartz, quartzite, sandstone, and sand crushed or disintegrated into sharp or round grains of uniform size.
For sawing and polishing marble, granite, etc.	Sharp and clean quartz graded into various sizes.
In sandpaper	Quartz, quartzite, flint, sandstone, and sand; coarsely ground and sized
Lithographers' graining sand	Medium to fine sand or coarsely ground silica, and tripoli.
Wood polishing and finishing	All forms of silica ground to medium fineness.
In scouring and polishing soaps and powders	Pumice, volcanic dust, quartz, quartzite, flint, chert, sandstone sand, tripoli, rottenstone, diatomite, feldspar, all in finely ground state.
Metal buffing, burnishing and polishing	Ground tripoli, and other forms of ground silica.
In tooth powders and pastes.	Various forms of pure silica, finely ground.

### DISTRIBUTION OF DEPOSITS

Although there are many deposits of sandstone, only a few are suitable for the production of grindstones. Carboniferous sandstones appear to be the most productive of the formations, as the producing quarries of the United States, eastern Canada, England, Germany, etc. are mainly of Carboniferous age. The thickness of the bedding as well as the closeness and uniformity of the cross jointing further determines the suitability of the sandstone. No matter how good the physical properties of the stone may be, the deposit is useless unless the beds are thick enough, and the joints far enough apart, to ensure that large grindstones or pulpstones can be cut and dressed from the quarried blocks.

*United States*

The bulk of the grindstones comes from the state of Ohio and a small amount from West Virginia; in the past it came from Michigan.

*Ohio.*—Sandstones from which the grindstones are obtained occur throughout the major portion of the state. The northern and western limits stretch easterly from the northeast corner of the state along and close to Lake Erie shores as far as Norwalk, 70 miles west of Cleveland, and thence south till they reach the Ohio River on the south.

The sandstones in Washington County are known as the Dunkard series and rank among the most important sources of grindstones. They are blue-gray to yellow, are of loose, medium-fine grits, being slightly finer than the celebrated stones from Newcastle, England, which otherwise they resemble. They are principally used for grinding-machine knives, saws, and thin steel tools. In the southern area the bulk of the present output of grindstones is maintained by four or five producing companies in the vicinity of Marietta and Constitution on the north bank of the Ohio River.

Berea, west of Cleveland, in the northern part of the state, is another center of the grindstone industry, there being numerous quarries within a radius of 15 miles. The stone is yellow-gray, of a coarse grit, and usually contains over 90 per cent silica. At present the principal quarries are at Amherst and Kipton, Lorain County and at Euclid, Cuyahoga County. Only a few grindstones are quarried at Berea, but raw material from many localities is sent there to be made up into grindstones.

The Amherst stone is buff, friable and soft. It is specked with iron, oxidized to limonite, which is loosely contained between the grains of quartz. The solid individual grindstone beds are about 8 ft. thick, with a total thickness of about 40 ft. The best stone occurs in huge lenses or "boulders," and sometimes several hundred feet long and 100 ft. thick. A fair production of grindstone is being maintained by one operator at Layland, Coshocton County. Other sandstones that have been quarried in the past are: the Euclid stone, a very hard, fine-grained, blue grit, which occurs in beds of about 20 in. and is used mainly as whetstones, or as a polisher for removing scratches made by the ordinary grindstones, and for grinding shoulders of knives; the Independence grit, which is coarse and sharp, used for spring and file grinding; the Massillon grit, from Stark County, similar to the Independence; the Tippecanoe grit, from Harrison County, which is fairly loose and hard and is used for grinding springs, files, and heavy forgings.

*West Virginia.*—Grindstones are now produced only to a small extent from the southerly extension of the Ohio sandstones of the Dunkard series in the vicinity of Ravenwood, Jackson County, and St. Mary's, Pleasant County. They are being worked by the Constitution Stone

Co., one of the principal producers of grindstones. (The company's main output is from Constitution, Ohio.) There are, however, many abandoned quarries on the south bank of the Ohio River. The sandstone occurs in blue and brown layers and in many places contains considerable mica.

*Michigan.*—Until comparatively recently, grindstones were produced at Grindstone City and Port Austin, in the Huron district, from fine-grained, uniform, blue-gray beds of sandstone containing numerous flakes of silvery mica. At one time the Huron grit was extensively used for grinding mowing-machine knives, cutlery and tools requiring a fine edge.

### *World*

*Canada.*—The quarrying and manufacture of grindstones is one of the oldest industries in Canada. In the Maritime Provinces there are upwards of a hundred old sandstone quarries which have at some time been worked for grindstones. The industry was at its height at the end of the last century, and has since steadily declined, owing originally to the opening of the Ohio and other quarries in the United States and latterly intensified by the introduction of manufactured grinding wheels. The main centers were on the New Brunswick and Nova Scotia shores of the Cumberland Basin off the Bay of Fundy and also along the south shore of the Bay of Chaleur and at Newcastle, N. B. The Read Stone Co., Sackville, N. B., has been in the grindstone business for almost a hundred years. At present it is the only producer, maintaining a small output from Stonehaven on the Bay of Chaleur, N. B., and from Quarry Island, Pictou County, N. S. Details of Canadian grindstone industry will be found in the Mines Branch report.<sup>25</sup>

*England.*—In the trade the stones of England appear to be roughly divided into three classes, referred to as: (1) Newcastle stones, (2) Derbyshire or Peak stones, and (3) Yorkshire stones. These designations, however, do not necessarily indicate the locality of origin of the stones.

The Newcastle grindstones are obtained from thick-bedded, light gray and yellowish sandstones of the Coal Measures at various localities near Newcastle-on-Tyne in the north of England. These stones are the most siliceous of the three classes. The rock is characterized by uniformity of texture, keenness of "bite," and compactness. The fine-grained varieties are in great demand for edge tools and glass beveling. For the latter purpose they are to some extent exported to America.

The principal producing centers are Springwell, Sheriff Hill, and Gateshead. Some of the largest and oldest quarries at Springwell, 3 miles from Newcastle, formerly owned by J. Elliot, were purchased during the latter part of 1924 by Richard Kell & Co., Ltd. These quarries are

stated to be among the best in the country for grindstones and pulpstones. Grindstones from 1 to 6 ft. or more in diameter are produced.

Derbyshire or Peak stones, which are coarse sandstones, are largely used for pulpstones. They are obtained from the Millstone grits, especially the Lower or Kinderscout grit. However, grindstones suitable for

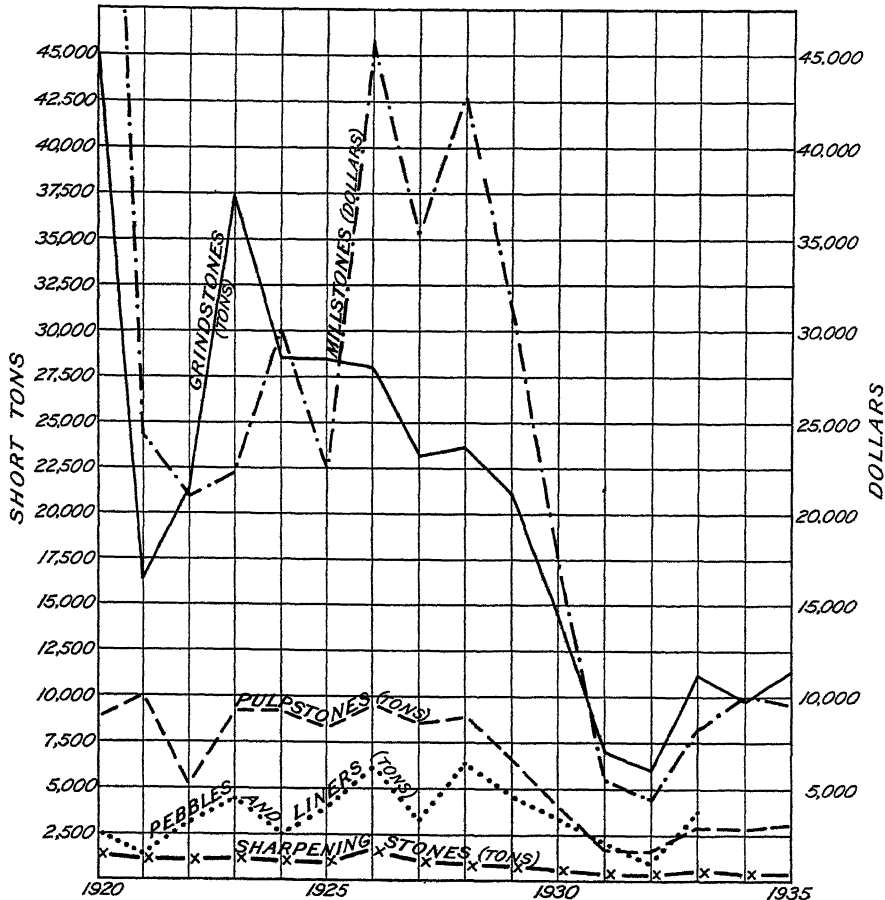


FIG. 4.—UNITED STATES PRODUCTION OF GRINDSTONES, PULPSTONES, SHARPENING STONES AND PEBBLES AND LINERS (TONS) AND MILLSTONES (DOLLARS), 1920-1935.

heavy work, such as grinding locomotive and heavy textile-machine parts, heavy edge tools, etc., are made from the coarsest Derbyshire stones. The principal producing centers are Rowsley and Matlock; the principal producer is James Akeroyd and Sons.

Yorkshire stones, typified by the Ackworth stone, generally have finer and sharper grits than the Derbyshire stones. They are usually light brown or buff, rather soft, fine-grained, thickly bedded sandstones. The best stones are obtained from the top of the middle Coal Measures.

The stones are used for grinding cutlery, saws, and fine edge tools. The grindstones are chiefly quarried from near Leeds, Pontefract, Rotherham, Normanton, Wickersley, Sheffield and at Cross Hills, the last named quarry being operated by Lombard & Co., of Boston, Mass.

*Other Countries.*—Details of the grindstone deposits and industry of other countries are lacking, but stones are being produced in Germany, Norway, Belgium, Italy and other central European countries.

### PRODUCTION

#### *United States*

The peak of production in the last 15-year period was in 1923; since then the output has steadily declined and is now almost one-third of that year (Fig. 4). Eight companies were active in 1936, the largest being the Constitution Stone Co. and the Cleveland Quarries Ltd. About 85 per cent of the total output of grindstones now comes from the Washington County quarries at Marietta and Constitution, Ohio.

#### *World*

*Canada.*—The grindstone output in Canada dropped from 2262 tons in 1920 to 360 tons in 1936.

*Other Countries.*—Production records of the larger producing countries such as England, Germany and Norway are not available, while statistics from other countries combine both grindstones and pulpstones. Some production and export figures are given in the Annual Statistical Summaries of the Imperial Institute, London, England.

### SPECIFICATIONS

A sandstone suitable for grinding purposes must be of uniform hardness, possess a sharp and even grain, and be free from clay or other impurities. The hardness is important, for if the stone is too soft it wears away rapidly, causing coarse and uneven grinding; if it is too hard it glazes and cuts so slowly that it is useless. The grains of silica should be cemented together in such a manner as to have sufficient tenacity to impart the necessary strength to the stone but at the same time crumble away with sufficient rapidity when exposed to friction to prevent glazing, as well as to present continually fresh, sharp grains. The size of the grit determines the ultimate use of the stone, as the coarser the grit the faster will be the cutting, although the work will be rougher.

### PRESENT STATUS OF THE INDUSTRY

During the latter part of the last century the grindstone industry was at its height, but for the past two or three decades there has been a gradual decline in tonnage production, caused mainly by the use of artificial

grinding wheels, which, owing to their superior cutting qualities, uniformity, ease of manipulation, and all-round efficiency, have replaced the natural stone for most general purposes. Today, the demand is mainly for stones of over 5 ft. in diameter, a market that limits the production to deposits of large, thick, and uniform beds. It is very doubtful whether grindstones will ever recapture the market held by the artificial abrasives for not only the practical but also the personal element must be considered. Workmen do not like to use grindstones, on account of the dirt and danger accompanying their use. At present the main uses for these large stones are, in order of their importance, for saw, file, machine-knife, scythe and shear grinding, for harvesting machinery and in the diemaking industry.

#### PULPSTONES

Pulpstones are made from sandstone having a somewhat similar texture to that used for grindstones. However, in rare instances the same quarry furnishes both kinds of stone, but always from different beds. Since the grinding face of the stone is at right angles to the bedding, the beds must be at least 3 ft. thick to make even the smallest pulpstones, and should be 10 ft., capable of yielding stones of 6 to 7-ft. face, which are now in the greater demand.

#### DISTRIBUTION OF DEPOSITS

The remarks regarding sandstone deposits suitable for grindstones are even more exacting for pulpstones, mainly because of the scarcity of deposits containing beds thick and free from cracks and inclusions from which the large stones can be produced. The United States and England are the foremost producers of pulpstones.

##### *United States*

In the past, pulpstones were produced from the sandstones at Steubenville, Lyria, Lisbon, Marietta and East Liverpool in Ohio, as well as from West Virginia and from Washington State. At present pulpstone activities are almost entirely confined to the sandstone beds along the Monongalia River and all within a radius of 10 miles between Fairmont and Morgantown in Monongalia County in the northern part of West Virginia. The quarries are at Opekiska, Flaggy Meadow, Tice and Uffington, and are now being operated by five companies, the Smallwood-Low Stone Co., West Virginia Pulpstone Corporation and the Smallwood Stone Co. being among the largest. On the west coast operations have been conducted for some years at Wilkeson, Pierce County, Washington, by the Walker Cut Stone Co., which in 1933 was one of the largest shippers of pulpstones.



*World*

*Canada.*—The principal sandstone beds from which Canadian pulpstones have, until recently, been obtained are in New Brunswick, in the vicinity of Newcastle and Quarryville, Northumberland County, and near Sackville and Dorchester, Westmorland County. The sandstone is somewhat soft, and beds thick and compact enough to supply the large stones are scarce. The present output comes from the west coast, on Gabriola and Newcastle Islands, off the east coast of Vancouver Island, British Columbia. The sandstone is harder and the beds thicker than those of New Brunswick. For full details see Mines Branch report.<sup>25</sup>

*England.*—The principal districts from which pulpstones are obtained are the close-grained, micaceous, drab sandstones of Darley Dale and the mottled coarser grained "Stoke" sandstone of Grindlefords, both in Derbyshire. Other districts are the yellow-brown beds of the Upper Coal Measures of Newcastle and Gateshead in Northumberland; also the Keighly district in Yorkshire and the Bilston quarries in Staffordshire.

*Other Countries.*—Pulpstones come from Sweden and Germany but information is lacking.

## COMMERCIAL CONTROL

Canada and the United States are large consumers of pulpstones and their production and sales are controlled by the situation of the pulp and paper industry. Some of these stones are exported to the American continent from England but competition is not serious. On the other hand, competition is increasing from the manufactured pulpstone produced in the United States and Canada. These stones are made up of segments of bonded silicon carbide or the fused-alumina abrasives. Well over 700 of these manufactured pulpstones are now in use throughout the world, over one-third of which are employed in Canadian mills.

## PRODUCTION

The United States and Canada are the only countries in which records are published separating grindstones, pulpstones, sharpening stones, and others.

*United States*

Pulpstone production is maintained from Monongalia County, West Virginia and from Wilkerson, Washington. The peak year was 1921, when 10,030 tons (\$750,063) was produced. There was a steady drop between 1928 and 1932; the output then increased slightly but is still less than one-third that of 1921. The production is shown in the general chart, Fig. 4.

The total imports by the United States of grindstones and pulpstones (not separated) in 1936 were 728 tons (\$24,638), England supplying 646 tons and Germany the remainder, against 534 tons (\$20,895) in 1935. The exports of these stones were 2480 tons (\$140,614) against 2764 tons (\$149,610) in 1935, of which in 1936 Canada took 90 per cent of the tonnage and 81 per cent of the value. Newfoundland took 1.5 per cent of the value. The remainder of the exports were principally small grindstones that went to 40 other countries, chiefly Mexico, South American countries and the West Indies.

*Canada.*—Pulpstones have been produced in Canada since 1892 and until 1923 all were from New Brunswick, the annual output ranging between 40 and 547 tons. In later years pulpstones have been obtained mainly from British Columbia. The peak production has been 1155 tons in 1926 and 911 tons in 1927; 266 tons in 1935 and 87 tons in 1936.

#### QUARRYING AND PREPARATION FOR MARKET

The flat bed of the rock from which the stone is to be made is cut out to the required dimensions, after which the block is removed, placed in a gang saw and cut to the approximate thickness. The squared block is then placed under circular saws; the external diameter is cut to within  $\frac{1}{2}$  in. of the required dimensions and the center hole is cut out by a small saw, after which the stone is sent to a semiautomatic finishing machine, which cuts the face and sides accurately. After the stone has been inspected the hole is bored out to the required shape and size, and the edges of the face are beveled to guard against accidental chipping.

The method employed by one of the largest pulpstone producers in the United States is as follows: The corners of the cubical block are broken off by means of small air hammers and the stone is worked into a more cylindrical shape by means of the same tool, the top face being roughly finished off. The stone is then turned over and the other face worked in the same way, after which the whole stone is gone over carefully with the air hammer. The eye is made by drilling nine holes through the center and reaming them out with an air hammer. It is then finally squared for mounting on the lathe shaft. For finishing, the stone is lined up in the square shaft and held in position by wedges, one end of the shaft being free and unsupported so that the stone can be quickly and easily removed. Finishing operations are similar to those already described under grindstones, the turning tool being a 6-ft. bar fitted with a forged drill-steel bit. The stone is made to revolve about 20 r.p.m. and each bit is renewed after 8 to 10 revolutions of the stone. The sides of the stone are finished first, then the face, and finally the corners, and after removal from the table the center hole is finished by air hammers using tools of different lengths.

In the quarry at Gabriola Island, Nanaimo, B. C., the stones are cut in place, by means of circular steel cylinders of exactly the same internal diameter as the finished pulpstones, and about 1 ft. longer. Each cutter is rotated on its vertical axis and is pushed downward by an automatic feeder, which regulates the rate of boring to about 2 ft. per hour (for a 64-in. by 6-ft. cylinder). Split steel shot is used as the cutting medium. The whole machine is mounted on wheels in a wooden frame, so that it can travel on rails on the wide benches of the rock. The surface of the rock immediately under the cutting edge is leveled with plaster of Paris before operations are begun. The stone cylinders are freed by drilling a number of small horizontal holes at the bottom of the bench and splitting by means of light charges of powder. They are then lifted by derrick, loaded into barges, and shipped to the McDonald Stone Co. at Vancouver, where the center holes are bored and the stones cut to the required length. It is claimed that the advantages over the cubical-block method are minimum waste of good stone, a saving of labor in trimming, and a considerable saving in freight charges.

#### SEASONING

The proper seasoning of pulpstones is of the utmost importance. The freshly quarried stone is saturated with water, which takes a considerable time to evaporate. As the moisture passes through the pores of the stone a process of cementation takes place, which forms a hard binder as the stone dries out. The finer the grit and the larger the stone, the longer the time required for drying out. The smaller stones should be seasoned for one year and the larger ones for about two years. In a poorly seasoned stone the cementation is not complete, the binder is soft and only the outside is hardened, consequently after a short time in operation the stone wears away rapidly or even fractures under the strain. The tying up of capital resulting from the stocking of a variety of stones valued at \$200 to \$1000 each presents a serious problem, and because of lack of capital, or rush orders, the length of proper seasoning is often not as long as it should be. Many experiments have been tried to shorten this period of seasoning by artificial means, but none appear to have been a real success. Among these is a process by which the stone is soaked in molten sulphur and allowed to cool, whereby the crushing strength is increased from two to three times the original. Although this is advantageous when the stone is to be used for building purposes, it does not appear to be successful for pulpstones. Another method of hastening seasoning is to steam-heat the stones to about 180° F. in a closed chamber for several hours and then allow them to cool slowly and age for about two months.

#### SPECIFICATIONS AND TESTS

*Specifications.*—A good pulpstone should cut fast, produce fibers neither too coarse nor too fine, be of medium hardness, have a long life,

wear evenly, and withstand the strain. In order to produce the long, thin fibers the stone should tear and not cut, hence sandstones composed of subangular grains are desirable. If the grains are too rounded there is a tendency for the stone to glaze, thus producing inferior fiber. Coarse-grained stones cut faster than finer grained, but the pulp from the former is coarse and of poorer quality. The bonding material holding the grains together should be soft enough to wear away so as to leave the harder silica grains protruding. The stone should be uniform throughout, otherwise it will wear unevenly. If too hard it is liable to glaze and require too frequent regrinding of the face; if too soft it wears away too quickly. The ideal pulpstone must give a satisfactory number of grinding hours under severe conditions; high daily production; long life—that is, installations must be kept low—and be of a quality that makes unnecessary too frequent dressing or “jigging” of the stone.

Sandstone deposits having beds of the requisite thickness and physical qualities are rare.

At one time pulpstones of 26 or 27-in. face and 54 in. in diameter were mainly used, but in the modern large mills the demand is for magazine grinders of 54 to 62-in. face.

*Tests.*—Although the small-scale tests on certain sandstones, to determine their suitability for the manufacture of pulpstones, do not duplicate the actual conditions in the mill, a comparison with standard pulpstones of good quality indicates to a large degree the suitability of the sandstone under trial.

Details of tests and results are given in the Canadian Mines Branch report,<sup>25</sup> but, briefly, these tests constitute granulometric analysis in which the sandstone sample is carefully broken down to its natural grain and the fineness ascertained by screening. The hardness is determined by grinding cores of the stone on a horizontally revolving plate upon which quartz sand is fed. The toughness is ascertained by subjecting the cores to an impact machine of the Page type and the height required by a pointed weight to crack the test piece is measured. The structure and nature of the grain and bond are determined by the microscope and also chemically.

#### MARKETING, USES AND PRICES

*Uses.*—Pulpstones are used for the production of wood fibers for papermaking. The previously cut short lengths varying from 2 to 4 ft. of barked wood are fed into a wood-grinding machine, where they are pressed by hydraulic pressure against the rotating face of the pulpstone.

*Marketing.*—In order to preserve the life and efficiency and obtain the full benefit of a pulpstone, it is necessary to treat it with care from the time it is cut out of the solid rock until it is finally discarded from the pulp mill. Great care must be exercised to prevent chipping of the face edges when handled by the cranes or if rolled over an uneven surface. It should be

crated for shipment and every endeavor should be made to eliminate the possibility of moisture and frost getting into the stone during transportation in winter. Whole carloads have not infrequently been ruined by neglect of the latter precaution. The rough handling of stones in quarry or transport often gives rise to the formation of fissures, which are the main causes of the breaking of a stone during operations.

*Prices.*—The values of shipments of all pulpstones from United States quarries averaged \$63 per ton for the past five years, the annual variations being from \$48 to \$83 per ton. In Canada there are almost 60 pulp mills using about 1000 stones valued at about one-half million dollars. The average value at the mills for the small stones for 2-ft. wood is \$300 each (2 to 2½ tons per stone); for 2½-ft. wood, \$335 each (3 to 4 tons per stone), and for 4-ft. wood, \$1100 (5½ to 7 tons per stone).

#### SHARPENING STONES

These small hand-operated stones include scythestones, whetstones, honestones, razor hones, oilstones, waterstones, holystones or rubbing stones. Their nomenclature is somewhat confusing, as the difference between these stones is not clearly marked and they grade from one into the other according to texture and hardness.

*Scythestones.*—This division, as its name implies, constitutes all stones used for sharpening scythes and sickles. The grit is also suitable for kitchen and butcher knives and similar articles. They are the coarsest of the whetstones and are usually made from the finer beds of sandstone from which grindstones are obtained. The better grades of scythestone are usually fine-grained, highly siliceous argillite or mica schist, which in many cases contain, in addition to the quartz, innumerable minute crystals of some hard mineral such as garnet or magnetite.

*Razor Hones.*—Razor hones embrace all stones used for sharpening razors and delicate instruments. At present razor hones are largely made from artificial abrasives, although there is still quite a demand for the natural Belgian honestone.

*Oilstones.*—These stones, as the name indicates, are those very fine grit stones from which the most satisfactory service is obtained by the use of oil. There is a big demand for artificial oilstones, although large quantities of the opalescent, siliceous novaculite from Arkansas are annually used.

*Waterstones.*—Water is used with these stones, although very satisfactory results are obtained with oil. The best varieties are the Hindostan stone from Indiana and the Queer Creek stone from Ohio.

*Holystones or Rubbing Stones.*—These stones, which are made from blocks of close-grained sandstone, were at one time extensively used for rubbing down ships' decks and the name "holystones" is said to have originated from the fact that the work was done in a kneeling position.

The stones are now used to some extent for rubbing down rough surfaces on which a fine finish is to be applied, particularly automobile bodies, furniture and concrete work.

## DISTRIBUTION OF DEPOSITS

### *United States*

Sharpening stones have been quarried and manufactured for many years in various parts of the United States. The following varieties are produced and are described under the type of rocks.

*Novaculite*.—Novaculite is a fine-grained and compact white, quartzose rock, supposed to be a consolidated siliceous slime. It consists almost entirely of chalcedonic silica (over 99 per cent) and is of sedimentary origin. The rock is quarried principally from the vicinity of Hot Springs, Garland County, Arkansas. There are two varieties, the true novaculite (Arkansas stone) which is white, and the Washita (Ouachita) stone, which in many instances has a yellowish or rusty red tint.

ARKANSAS STONE is very dense and has a conchoidal fracture and waxy luster. It occurs in massive beds a few inches to 15 ft. thick, in the novaculite formation, which has a total thickness of 500 ft. and includes thin layers of shales and sandstones. As a result of pressure, the brittle novaculite has in many places been crushed and split; and, in addition, many fine quartz veins intersect the rock in all directions, some so thin as to be invisible to the naked eye. As these are detrimental, careful sorting is necessary, and the amount of waste is out of all proportion to the material eventually used. Large blocks of even apparently pure stone are not shipped, but are broken into pieces weighing up to 5 lb. Freezing also has an injurious effect on this stone, as its denseness does not allow for expansion. The Arkansas stone, which is produced in "hard" or "soft" grades, is especially suitable for sharpening fine-edged instruments and small tools and is used mainly by surgeons, dentists, engravers, jewellers, et cetera.

WASHITA STONE has approximately the same composition as the Arkansas but is less dense and more porous, has a subconchoidal fracture and resembles unglazed porcelain. It is freer from defects than the Arkansas stone; quartz veins are not so numerous, but cavities or "sand holes" are much more common. On account of its greater porosity, freezing does not have an injurious effect. Long drying is inadvisable, however, as it seems to cause loss of easy fracture and to make the stone tough and hard. The stone is less expensive than the Arkansas and is a faster cutting stone, but is used for coarser work. The Washita stone is shipped in blocks weighing 50 to 1000 lb. It is found in much larger quantity than the Arkansas stone and the demand for it is also much

greater. It is claimed that these novaculites give a much smoother and longer lasting edge than do the artificial stones.

*Mica Schist.*—Fine-grained, dark gray, highly siliceous, laminated mica schist is quarried at Pike, Grafton County, N. H. The quartz grains occur in definite layers separated by thin layers of mica. The part of the rock containing coarse, irregularly scattered quartz and argillaceous matter is discarded. There is a regular system of cross-jointing at right angles to the bedding planes, so that after blasting slabs or "bents" are easily pried out. The irregular slabs are first roughly trimmed, then further reduced and stacked. The Pike quarries have been producing scythestones for over a hundred years.

At Lisbon, also in Grafton County, the Pike company at one time produced the "chocolate" stone from a bluish chocolate colored schist. The demand for this stone has died out, so that quarrying for a few weeks provides enough for several years. It is used mainly by fishermen along the coast. In a quarry at Evansville, Orleans County, Vt., the rock is of a more massive formation than the Pike, and does not occur in layers.

*Sandstone.*—Among the better known is the Hindostan, or Orange stone, from French Lick and Northwest Township, Orange County, Indiana. Some of the crude material is shipped to Pike or Littleton for manufacture. It is a fairly soft, fine-grained, low-priced waterstone used mainly by mechanics and for domestic service. West Baden, Ind., is also a producing center.

Scythestones have been manufactured by several of the Ohio grindstone companies, particularly from the Berea and Euclid grits. The Queer Creek stone is a hard, dark gray, medium coarse sandstone, which is used as a waterstone for coarse sharpening. The Euclid is a blue, very fine grit stone used mainly for butcher and pocket knives. The Berea grits are used for the coarsest work, and the Huron for medium.

The Deerlick stone at Chagvin Falls, Cuyahoga County, Ohio, and the Labrador stone, which is somewhat similar to the Ohio sandstones, near Labrador Lake, Cortland County, New York, were at one time quarried to a small extent.

*RUBBING STONES.*—There are a number of sandstones of various grits, known as brown, blue, and red, that are extensively used for honing or rubbing marble or granite by hand. Other types of compact micaceous stone used for this purpose are the American Black Hone, and the Kentucky Caron Hone from Louisville and from West Baden, Indiana.

*Operators.*—During 1936 there were ten operators. The Norton-Pike Co. (the Pike Manufacturing Co. and the Norton Company amalgamated in 1933) being by far the largest. It produces scythestones from the schists of Pike, N. H., and Evansville, Orleans County, Vt., and also oilstones from the novaculite at Hot Springs, Ark., mined under contract. During the year scythestones were also made from the sandstone at

Berea, Ohio, and at West Baden, Ind., also rubbing stones from the latter district.

*Artificial Stone.*—Artificial sharpening stone made up of bonded fused alumina or silicon carbide fine grit is becoming more in demand than natural stone because of its faster cutting. It is probable, however, that there will always be a demand for natural novaculite oilstone for tools requiring a very keen edge, and for sandstone products for tools requiring a coarse, rough edge.

### World

*Canada.*—There are many fine-grained sandstones, siliceous argillites and mica schists in Canada that are suitable for sharpening stones, particularly in the Eastern Townships of Quebec; in Cumberland and Pictou Counties, Nova Scotia, and at Chaleur Bay, New Brunswick. A small output is maintained for fine-grained, blue-gray sandstone at Stonehaven, on Chaleur Bay, and from Shediac, Westmorland County, N. B.

*United Kingdom.*—Scythestones and honestones are made from the finer-grained sandstones of Bilston, Newcastle and from several parts of Yorkshire. A fine-grained micaceous sandstone from Talacre, Flintshire, is used for scythestones.

The most important kind of whetstone is the Scotch hone, sometimes called Water-of-Ayr, Snakstone, or Tam O'Shanter, obtained from Bridge of Stair, near Dalmore, Ayrshire, Scotland. It is a mottled, pale gray, very compact, Carboniferous shale and is used as an ordinary whetstone, also for a lithographic stone and for burnishing pulpstones.

Another good hone is the Charley Forest or Whittle Hill stone from Leicestershire, an exceedingly fine-grained, hard tuff from the Blackbrook series. The Idwal or Welsh oilstone, and the Cutler's Green from Snowden, Wales, have also been extensively used. The so-called "rag-stones" obtained from Scotland, and elsewhere, consist of siliceous mica schists having a twist along their length which gives a sharp "bite."

*Belgium.*—The well-known Belgian razor hone, sometimes called "soapstone" or "petrified woodstone," is a damourite slate containing innumerable garnets. The stone occurs in the form of 2 to 3-in., white to yellowish bands in a blue-gray slate (phyllade). As the slate has abrasive qualities also, the stones are usually double-faced, with the yellow damourite on one side and the gray slate on the other. These hones are quarried at Lierraux, Salm-Chateau, Bihau Sart, and Recht, in the Belgian Ardennes.

*Germany.*—The German water hone from Sonneberg is a hard, fine-grained, bluish green argillaceous schist. The Ratisbon hone, which is similar to the Belgian stone described above, occurs as a yellowish band from 1 to 18 in. wide in a blue slate.



*Asiatic Turkey.*—The oilstone from Smyrna, in Asia Minor, is in nearly all respects similar to the Arkansas novaculite, but it is not so uniform. It is, however, drab in color and carries an appreciable amount of free calcium carbonate and other impurities. Before the discovery of the Arkansas stone, it was regarded for several centuries as the best oilstone for mechanical tools.

*Other Countries.*—Whetstones are also made in Bavaria, Italy, Sweden, France and Japan.

### PRODUCTION

*United States.*—The present sources of supply are briefly reviewed under Distribution of Deposits. Statistics are given in Fig. 4. The peak of production during the past 15 years was 1680 tons (\$223,359) in 1926, but in the past five years production has been under 600 tons, the 1936 output being 752 tons (\$121,196). During 1935 some competition was felt from imports of natural sharpening stones, which amounted to 101 tons (\$53,563), of which 47 tons (\$4052) were from Germany; 24 tons (\$40,679 from Great Britain and 17 tons (\$1241) from Japan.

*Canada.*—From 50 to 100 years ago the output of these stones was substantial, but for the past 15 years the annual production has ranged between 17 and 169 tons, the 1936 output being 129 tons (\$4872). The low value is because the material from Shediac, N. B. is shipped in crude blocks to a United States manufacturer for finishing.

*Other Countries.*—Statistics of most of the other producing countries are not published separately, though France recorded 1120 long tons in 1928; 3320 tons in 1929 and 200 tons in 1932; Italy's records, however, from 1928 to 1933 show the following: 1796, 2377, 1568, 1500, 1250 and 1401 long tons of sharpening stones and whetstones.

### PREPARATIONS FOR MARKET AND USES

*Scythestones.*—For the making of scythestones from the fine-grained, siliceous schists of New Hampshire and Vermont, the rough slabs or "timbers" of the selected material are split at the quarry by means of knives and hammers, into approximately the required shape. They are then conveyed to the mill and placed on the rubbing beds, where they are ground to a uniform size. They are reversed until the required thickness is obtained, then are done up in bundles of 50 and the ends are trimmed to the required length. Each stone is finished separately by hand on the bed, the edges and ends being beveled. They are finally smoothed off by hand by means of a piece of hard stone, then are labeled and packed in boxes. Local sand is used as the abrasive to grind the stones and is fed to the bed by means of a Frenier pump.

*Arkansas Oilstones.*—The Arkansas novaculite from Hot Springs and the more porous Washita stone come from the mines in relatively

small lumps. The lumps are built up in layers into a large block, using plaster of Paris as the cement. When dry, the whole mass is sawed vertically by band saws into 2-in. slabs, after which the block is then sawed in a horizontal direction. The separate long strips are laid flat and again built up into a block with plaster and cut by the saws to the required length of about 8 in. The finishing is done by laying the pieces, after they are freed from the plaster, on the revolving face of a horizontal iron wheel, 5 to 6 ft. in diameter, using sand as the abrasive. The water absorbed by the Washita stone during the sawing or rubbing processes gives it a bluish green color, which is considered objectionable, so that after drying the stones are rubbed or "cleaned" with pumice to restore the original white.

The discards and small slabs resulting from the sawing operations are set evenly with plaster of Paris on the face of a small disk, which is placed face downwards and rests on the rubbing wheel. There are four of these disks to one wheel. Both the rubbing wheel and the disks containing the cemented stones revolve, resulting in a double grinding action. The process is repeated for the reverse side and for the ends. These disks and the small odd pieces are made up into the numerous forms and shapes requisite for the trade. Thin disks of novaculite used by jewelers, dentists, etc., are cut by diamond-toothed cylinders. Any other pieces that are not made up are ground into a powder and used by makers of watches and razor blades for polishing and grinding.

*Manufactured Sharpening Stones.*—For some purposes sharpening stones are made up artificially from a mixture of natural grits, such as a fine sand, crushed siliceous schist, and a clay bond. The ingredients in certain proportions and size of grit are thoroughly mixed together with water and silicate of soda, pressed into various shaped molds and baked. A typical stone of this kind is fitted with a handle and used for domestic sharpening of knives.

*Uses.*—The uses for sharpening stones are indicated under the description of the rocks from which they are made, the sharpening of coarse, medium or fine-edged tools being governed by the type and texture of the stone.

## MILLSTONES

The term "millstone," which includes the true burrstone and the chaser stone, is somewhat loosely applied to include circular stones revolved on a horizontal plane as well as those run on edge. They may be made from any hard and suitable rock varying from a sandstone, basalt, granite, to a quartz conglomerate. The true burrstone is a white to gray or slightly yellowish form of chalcedonic silica. The stone has a coarse, cellular structure, the holes being frequently due to the dissolving out of calcareous fossils. It has about the same hardness as flint, but is

not so brittle and possesses a straight fracture. Its sharp cutting power is due to the toughness of the rock and its numerous pores and cavities.

#### DISTRIBUTION OF DEPOSITS AND PRODUCTION

Rocks suitable for millstones occur in many countries and at one time the volume of trade in these stones was large. The United States, France, Sweden, Greece, Turkey, Yugoslavia, Italy and several other European countries are manufacturing millstones from beds of different kinds of rocks.

*United States.*—About 40 or 50 years ago millstones valued at around \$100,000 were produced annually in the United States, but recently the output has been between \$5000 and \$10,000 (Fig. 4).

Millstones have been produced from a fine-grained quartzite at Brush Mountain, Montgomery County, Virginia; from a quartz conglomerate at Parkewood, Moore County, and from a granite at Faith in Rowan County, both in North Carolina; from a quartz conglomerate, Lancaster County, Pennsylvania; from the Dutton sandstones of Jackson County; from a quartz conglomerate of similar texture to the Esopus stone, near Fair Haven, Rutland County, Vt.; and from a white variety of the Berea sandstone at Peninsula, Summit County, Ohio.<sup>26</sup> They are now made from the Shawangunk quartz conglomerate by four operators at Accord, two at Kerhonkson, and one at High Falls, Ulster County, N. Y.; one at Salisbury, Rowan County, N. C.; one at Cambria and one at Blacksburg, Montgomery County, Va. Import of millstones into the United States is very small; that in 1936 was 22 tons (\$2228), all from France.

*Canada.*—When the consumption of millstones was heavy, some 40 or 50 years ago, they were produced from Millstone Island, near the south-east extremity of Guysborough County, Nova Scotia, also from the Chaudiere River, Quebec; from north of the Cascades; at Murray Bay, Quebec and from near Cayuga, Ontario. It is, however, over 30 years since any millstones were produced in Canada.

*France.*—Some of the best burrstone is produced in France. It is a fresh-water, cellular quartzite or flint of Tertiary age, having great strength and toughness. The best stone comes from the top of the lower Oligocene of the Paris Basin, from a bed called the "Calcaire de Brie," which stretches from Vernon to Rheims and from Laon to Fontainebleau. The principal quarries are at La Fertesous-Jouarre. The stone occurs in large masses and is worked in open quarries and usually sold in irregular blocks. The blocks are shaped to the required dimensions and sold as solid stones, or else fitted together and bonded into solid wheels. The stone finds markets all over the world, and in 1922 about 434,000 short tons of burrstones and millstones was produced, but in 1932 this had dwindled to 23,200 tons.

*Germany.*—Millstones are produced from several localities in Germany, the principal one being at Andernach, 10 miles northwest of Coblenz, where the Niedermendig stone is mined. This is a porous trachyte-tuff. Other millstones are obtained from porous rhyolites found in the Odenwald, Thuringia, and the Fichtelgebirge district. The Ziltan millstone is a coarse conglomerate in a sandy matrix obtained from south-east Saxony.

*Other Countries.*—The number of stones produced in Belgium in 1929 was 141,700 and in 1933 was 51,010. The number in Greece was 1893 in 1929 and 4232 in 1933, and Turkey during the same years produced 2740 and 2768 millstones, respectively.

#### CHASER STONES

Chaser stones are large, circular stones, run on edge, usually in pans paved with blocks of conglomerate or some hard material, and are used mainly for grinding minerals, chiefly feldspar, quartz and barites. They are made from a rock similar to those from which millstones are produced, but being larger are used for heavier work.

In the English chaser mills used for grinding spices, both Cornish and Aberdeen granites are employed; probably many other local igneous rocks could be so used, but lately it has been found more economical to import Italian granite for this purpose. In the Staffordshire potteries, two distinct varieties of chert were employed in grinding mills, the lower stone being a chert obtained from the base of the Millstone grit at Halkin Mountain, Flintshire, Wales. The upper was a cherty limestone obtained from Bakewell and Longstone Edge, Derbyshire.

#### GRINDING PEBBLES AND TUBE-MILL LINERS

Extremely hard pebbles of flint or quartzite were used at one time to a large extent in cylindrical or conical mills for the grinding of cement, ores, paint, soft minerals, clays, etc. Steel balls and steel or rubber liners have to a large extent replaced the natural products, except where there may be danger of iron contamination from metal worn off the balls or liners. On this account the pottery industry is now probably the largest consumer of natural pebbles and liners.

In the United States the largest producer of both pebbles and liners is the Jasper Stone Co., Sioux City, Iowa. Small cubes or blocks are cut from the quartzite at Jasper, Minn., and artificially rounded by tumbling in rotating cylinders. Liners are cut from the same rock. These pebbles and liners are used for grinding cement, ores, silica, paint, gypsum, feldspar and in the ceramic industry. A few car lots of beach pebbles are shipped annually from Encinitas Beach, near Carlsbad, San Diego, Calif. These pebbles of granite, porphyry and quartzite are continually being carried ashore by means of floating kelp plants, to which they are

attached. The production in the United States during the past 15 years has ranged between 2000 and 6300 tons, except for the low year of 1932, when only 976 tons was sold (Fig. 4). The average value is about \$13 per ton.

In Canada, granite pebbles used for clinker grinding and in cement mills were at one time produced from the vicinity of Santoy, near Jackfish, on the north shore of Lake Superior, but none have been shipped for the past few years. Other localities are at Hedley, B. C.; Gabarus Bay, Cape Breton County, Nova Scotia, and in the Cypress hills of Saskatchewan.

The best known flint pebbles are obtained from deposits in Denmark, Belgium, and from the sea coast of France between Havre and St. Valéry-sur-Somme. Danish pebbles are recognized as the standard, because of their great hardness and toughness.

Flint pebbles are also produced in England and Norway; and various types of grinding pebbles have been exported from Germany, Italy, Labrador, Newfoundland and Japan. Belgium and Sweden are the chief foreign sources of tube-mill liners.

Imports of these products into the United States in 1935 amounted to 7829 tons (\$66,727) of which about half were from Denmark and the remainder from France and from Belgium.

There are seven sizes of Danish pebbles, ranging from 1 to 8 in. in diameter, each size from No. 1 to No. 7 being about 1 in. larger than the preceding one.

### SILICA

Silica sand and quartz are reviewed in detail in chapter XLI, so that only their abrasive uses and applications are given below.

*Silica Sand.*—Silica sand is extensively used for sandblasting purposes, for the initial grinding or surfacing of plate glass, and as a cutting medium for gang saws on stone.

For sandblasting the main centers of production in the United States are Ottawa, Ill., and Cape May, N. J. The Ottawa material occurs as a friable sandstone, which is broken down to its natural grain, washed, and screened. The grains are spherical, resembling miniature pearls when seen under the lens. The Cape May sand is subangular and uneven in shape, but larger grain sizes are obtainable, more so than with the Ottawa sand.

In Canada sandblast sand is obtained from the decomposed rock containing friable quartz and china clay at Lac Remi, north of Montreal, Que.; from a similar type of material near Smoky Falls, north of Lake Nipissing, 25 miles west of North Bay, Ontario; also from a friable quartzite at East Templeton, Que., a few miles northeast of Ottawa, Ont. The grain of the Canadian sands is sharp.

The ranges of grain size are approximately: No. 1, between 20 and 35 mesh for light work; No. 2 between 10 and 28 mesh; No. 3 between 6 and 10 and No. 4 between 4 and 8 mesh. The last two are used for the heavy cast-iron work and steelwork.

Pure, clean beach and river sands, or Illinois sand, are used for the preliminary or coarse surfacing of plate glass. The crushed sand is water-graded into a number of grades at the glass plants and fed to the surfacing machines. Approximately three tons of sand is required to surface one ton of plate glass.

Cutting sand, composed of sharp, solid, quartz grains, is used as the abrasive for sawing stone. It is usually ungraded and about equivalent to a No. 1 sandblasting sand.

Burnishing sand is a fine, rounded-grain silica sand and of uniform size between 65 and 100 mesh, used in rolling down and burnishing gold decorations on porcelain. Tests on sandblasting sands and an interesting discussion on round versus sharp grains are given in detail in L. H. Cole's report.<sup>24</sup>

*Quartz*.—Crushed and graded quartz is used for the abrasive backing of "flint" sandpapers. Almost any deposit of massive white quartz is suitable. Being the cheapest of all the abrasive-coated papers, it still is sold in fair amount, mainly in hardware stores and by small jobbers. It is made only in the form of paper, not as cloth. True chalk flint from England and France is extensively employed for this purpose in Europe and has better cutting qualities and longer life than ordinary quartz.

Powdered quartz and silt are sometimes used for scouring compounds and for the harsher metal polishes.

#### SOFT SILICEOUS POWDER ABRASIVES

Many natural highly siliceous materials either occur as a powder or are used only in the powder form for mild abrasives. For the majority of these use as an abrasive is of minor importance as compared to their principal applications. Among these, diatomite, pumice and tripoli are reviewed elsewhere (chapters XIII, XXXV and XLVIII). Their abrasive uses are therefore only briefly given below.

*Pumice*.—Under this title are included lump pumice and pumicite or volcanic dust, the natural powder. Lump pumice is used by manufacturers of furniture and musical instruments for dressing the wood and metal surfaces; by silver platers for preparing their metal surfaces; by lithographers for cleaning the stone surfaces; for rubbing down and polishing fine tools and instruments and for domestic and toilet uses, such as hand cleaners. Pumicite or ground pumice is mainly used as a cleanser, the thin, sharp and striated grains being particularly suitable for this purpose. It is also made up in the form of hand soaps. During 1935, in

the United States, 50,640 tons valued at \$176,456, or 88 per cent of the total tonnage of pumice sales, was used for cleansing and scouring compounds and for hand soaps, and about 2 per cent for other abrasive uses, which include polishing compounds, polishing powders for bone, celluloid, and hard rubber, in dentists' tape and in some rubber erasers.

An artificial pumice used for abrasive purposes is made in Germany. In this process obsidian or volcanic glass preheated to 900° C. is dropped down a shaft furnace against a rising draft of hot gases. The obsidian swells into porous particles and is then consolidated with lime or cement to a form resembling a pumice, which has an apparent specific gravity of less than that of natural pumice.

*Tripoli, Microcrystalline Silica and Rottenstone.*—These fine-grained, porous materials are known to the trade as "soft silicas." Tripoli, which in the United States comes from southwest Missouri and northeast Oklahoma, is mainly used (about 70 per cent of tripoli production) in the form of made-up tripoli grease bricks or tripoli compositions for buffing and polishing. The compound is applied to a rapidly revolving belt or canvas wheel and used for the finishing or buffing of metals, plated products, etc. It is also used to a small extent in the manufacture of some scouring and cleaning powders and soaps; for the rubbing down of painted surfaces, such as automobile bodies. A similar but finer grained material occurring in the northwest corner of Arkansas, about 50 miles southeast of the Missouri deposits, is used mainly for oil-well drilling mud. Microcrystalline (sometimes erroneously termed "amorphous") silica, which comes mainly from southwestern Illinois, and to some extent from Wayne County, Tennessee, is also used as buffing and polishing compounds. These compounds are termed "silica" by the trade and are much in demand for white "coloring" operations on high-class work. Chemically precipitated amorphous silicas are also used in polishing and buffing compositions. Both tripoli and microcrystalline silica are mined from deposits at Harrisburg, northwestern Georgia. Rottenstone, a fine-grained gray-buff siliceous-argillaceous limestone, comes from Antes Forte, Lycoming County, Pa., and is used as a polish base; for instance, for automobile polishes. During 1936, in the United States, 16,443 tons valued at \$247,948, which was 59.8 per cent of the tonnage and 64.0 per cent of the value of the total sales of these three materials was used for abrasive purposes, mainly in grease compositions.

*Diatomite.*—Approximately 98 per cent of the diatomite (diatomaceous silica, kieselguhr, etc.) production in the United States comes from the Marine deposits of western California. The amount used for abrasives is insignificant in comparison to its other applications. As an abrasive it is mainly used in metal (silver) polishes, powders and pastes and in some automobile polishes, dental powders and pastes and occasionally as a friction agent in match heads or on the sides of boxes.

## NONSILICEOUS SOFT ABRASIVES

*Ground Feldspar* is employed to some extent in scouring and cleaning compounds and for a window cleaner.

*Lime*.—This so-called “Vienna” lime (originally from Vienna, Austria) that is used on the American continent in grease-brick buffing composition is obtained from certain beds of dolomite at Francis Creek and Manitowoc, Wis. Vienna lime is made by calcining the dolomite and cleaning and grinding to a certain fineness. It is packed in carefully sealed containers. As soon as the lime becomes hydrated it ceases to function and also attacks the grease compositions. It is used for the buffing of brass, copper, bronze, steel, pearl, celluloid, etc., but its main use is for the “coloring” of nickel after plating, as it gives it a deep “under surface” blue peculiar to the metal. Lime attacks aluminum, therefore it is not used on that metal.

*Chalk*.—Chalk (calcium carbonate) is a soft, compact, fine-grained, white limestone composed of the calcareous remains of small Marine shells. A small amount of this chalk—mainly from England and France—known as “whiting” is used as a very mild abrasive for hand-polishing of nickel, gold, silver or plate ware, buttons, et cetera.

*China Clay* (kaolin) and some pipe clays have been successfully used in polishing powders. Pipe clay was at one time the standard polish for naval and military tunic buttons.

*Talc* is used for polishing rice grains.

*Metallic Oxide Buffing Materials*.—As these are manufactured products, they are outside the sphere of this review. Briefly, they consist of various iron oxides such as crocus (red-brown), rouge (red), black rouge (magnetic iron oxide) mainly for glass, green rouge (chromium oxide) mainly for platinum and stainless steels; satin rouge (lampblack) for celluloid and bone; manganese dioxide; putty powder (tin oxide) for stone polishing.

## MANUFACTURED ABRASIVES

Manufactured or artificial abrasives are divided into four main groups: (1) silicon carbide (sold under the trade names Carborundum, Crystolon, Carbolon, etc.); (2) fused alumina (sold under the trade names Alundum, Aloxite, Lionite, etc.); (3) boron carbide (Norbide); (4) metallic abrasives such as steel shot and grit and steel wool, and various metallic oxides used in buffing and polishing compounds. As these are all manufactured materials, rather than natural mineral products, space is devoted to them only to the extent of showing the amounts in which they are used in competition with the natural abrasives. Details of their manufacture and uses are given in the Mines Branch publication.<sup>25</sup>

Silicon carbide is made by three companies having one plant in the United States and three plants in Canada. Fused-alumina abrasive is



made by six companies having two plants in the United States and five plants in Canada. The three manufacturers of silicon carbide make both products and are included in the fused-alumina list. There are also manufacturers of both products in France, Norway, Sweden, Germany and other European countries.

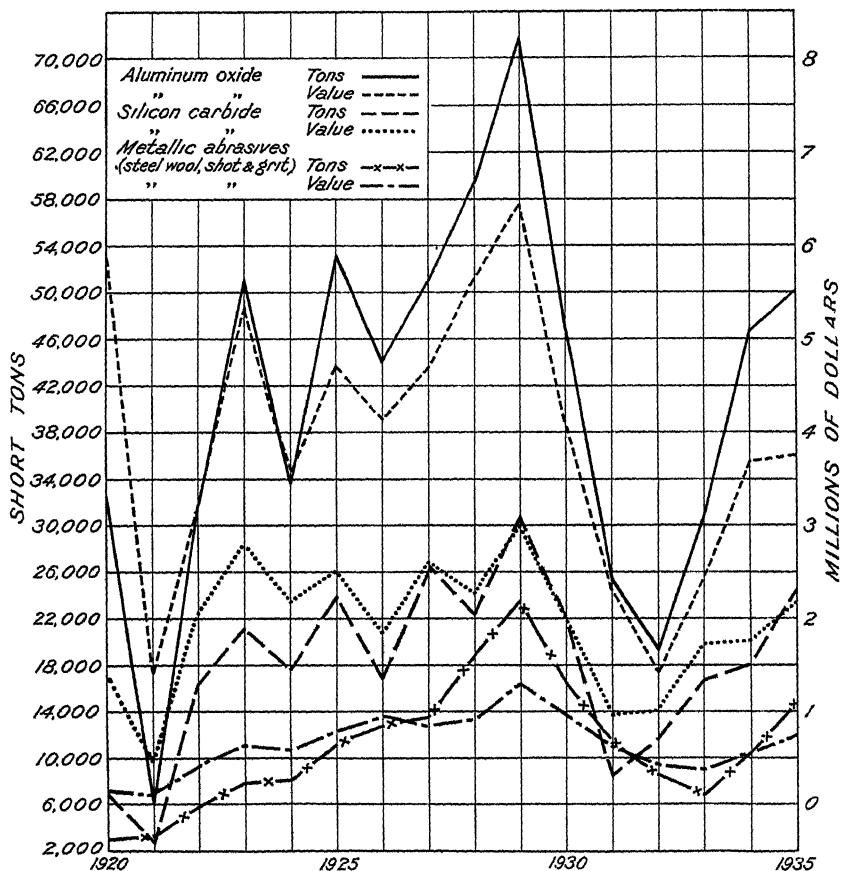


FIG. 5.—CRUDE MANUFACTURED ABRASIVES SOLD OR USED FROM PLANTS IN THE UNITED STATES AND CANADA, 1920-1935.

The new product Norbide (boron carbide) was produced by the Norton Company, Chippawa, Ont., Canada, in 1934. This product, harder than silicon carbide or fused alumina, is the hardest known manufactured material, very closely approaching the diamond. Its uses are now being developed, but at present its main outlet is in the form of molded products for sandblast nozzle liners, extrusion dies and for thread guides in textile machinery. The powder is used for many grinding and lapping operations that previously have been possible only with diamond dust. It is of interest to users of hard abrasives that the old Mohs' scale, in which quartz was 7, garnet 8, corundum (fused alumina) 9, and diamond 10,

has been extended to quartz 8, garnet 10, corundum and tungsten carbide both 12, silicon carbide 13, boron carbide 14, and diamond 15.

The large volume of business in recent years of these manufactured products is indicated by Fig. 5. In the peak year of 1929 the output from United States and Canada of silicon carbide, aluminum oxide and metallic abrasives amounted to 126,712 tons, valued at \$10,821,696, after which there was a substantial drop up to 1932, when the value was under three million dollars, and then a gradual rise to 123,834 tons (\$7,274,986) in 1936.

Approximately 95 to 98 per cent of the total imports into the United States of silicon carbide and fused-alumina abrasives are from Canada in the form of crude material made therein by companies of which the head offices and crushing and grading plants are in the United States. From 1000 to 1500 tons of the manufactured abrasives are imported annually from Europe and come from Norway, Germany, Sweden, Austria and France.

Details covering the manufacture of these abrasives, as well as the manufacture of grinding wheels, sandpaper, buffing compounds, etc., and their special industrial applications will be found in the Canadian Mines Branch report.<sup>25</sup>

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## CHAPTER II

### SILLIMANITE GROUP—ANDALUSITE, KYANITE, SILLIMANITE, DUMORTIERITE

BY PAUL F. KERR,\* MEMBER A.I.M.E.

**R**EFRACTORIES as a whole are treated in a general chapter by Tyler and Heuer. In this and other chapters the special characteristics of individual minerals of particular importance in their field are discussed more in detail. Andalusite, kyanite, sillimanite and dumortierite† have in common the capacity to withstand high temperatures and to change over into mullite, and it is this that makes them useful as refractories.

The four minerals andalusite, kyanite, sillimanite and dumortierite may be considered as a group because of similar ceramic use. All are in demand for high-grade refractories. Porcelain produced from these minerals is valuable because of its high melting point, high insulating power when heated, low coefficient of expansion and resistance to the corrosive action of certain fluxing agencies and furnace gases.<sup>59</sup>‡ The minerals of this group toughen the product to which they are added and are favored in the manufacture of porcelain for spark plugs, refractory brick, electrical and chemical porcelain and chinaware, enamelware and hotelware. Refractories with these minerals as component parts are used in glasshouse refractories; crucible, boiler, brass-melting, electric and forging furnaces; high-temperature cements; oil and gas fireboxes; combustion tunnels and cement-kiln linings.

#### COMPOSITION

The three aluminum silicates, andalusite, kyanite and sillimanite, have an  $\text{Al}_2\text{O}_3:\text{SiO}_2$  ratio of 1:1, but differ in crystallization. Andalusite and sillimanite are orthorhombic, but structurally different, while kyanite is triclinic. Dumortierite is a basic aluminum borosilicate,<sup>42,56</sup> approxi-

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† Synonyms or varieties:

Andalusite var. *chiastolite* is a type found in crystals having symmetrical black markings due to carbonaceous inclusions. *Viridine* contains iron and manganese (mangan-andalusite).

Sillimanite syn. *fibrolite* var. *monrolite*, *bamlite*, *zenolite* and *worthite*.

Kyanite syn. *cyanite*, *disthene*. (Committee on Nomenclature, Mineralogical Society of America has approved the spelling *kyanite*.)

‡ References are at the end of the chapter.

mately  $8\text{Al}_2\text{O}_3 \cdot \text{B}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot \text{H}_2\text{O}$  and orthorhombic in crystallization. It is closely related in thermal behavior to the three aluminum silicates mentioned and has a corresponding practical application. The four minerals are converted above  $1545^\circ \text{C.}$  to an artificial product<sup>2</sup> having the composition  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  and vitreous silica. The aluminum silicate,  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ , is generally referred to under the name mullite because of agreement with the rare mineral of that name. The vitreous silica is thought to be cristobalite.<sup>14</sup> In recent years outstanding contributions have been made to the understanding of the binary series alumina-silica.<sup>2,45</sup> Bowen and Greig have shown that the constituents resulting from the fusion of pure  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  in all proportions and at temperatures up to  $2000^\circ \text{C.}$  are liquid, cristobalite, mullite and corundum (Fig. 1). The

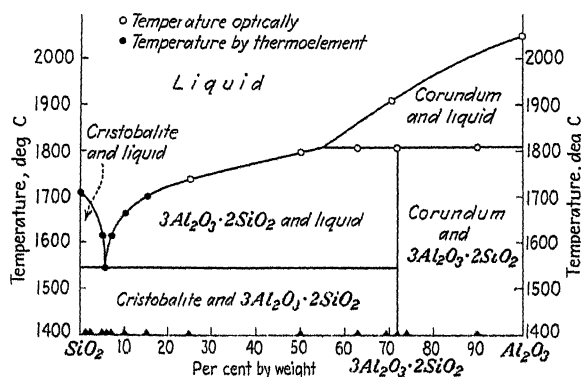


FIG. 1.—EQUILIBRIUM DIAGRAM OF THE SYSTEM  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$ , AFTER BOWEN AND GREIG.

mixture 71.8 per cent  $\text{Al}_2\text{O}_3$  and 28.2 per cent  $\text{SiO}_2$  forms pure mullite, which is stable to  $1810^\circ \text{C.}$ ; above this temperature it forms corundum and liquid.<sup>3</sup> Mullite<sup>3</sup> is orthorhombic in crystallization with a slight but definite difference from sillimanite. This difference was not detected at first even with X-ray work,<sup>5,31,32,33,60</sup> and early descriptions refer to the artificial product secured by calcination of the aluminum minerals as "artificial sillimanite."

#### PROPERTIES

The ordinary physical properties and the optical properties of the minerals belonging to this group are outlined in Table 2. The optical properties<sup>8,29</sup> are probably most used in identifying the minerals, and specific gravity is relied upon to a large degree in estimating commercial grades. The thermal properties of the minerals are of primary importance in determining their usefulness in industry. The utilization of all these minerals depends upon the inherent properties of mullite and the type of mullite crystals produced by calcination. Mullite is heat-resistant, is a good insulator even at high temperatures<sup>28</sup> and, perhaps

most important of all, is resistant to shock.<sup>41</sup> Mullite remains stable to a temperature of 1810° C. Above this temperature it does not break down completely but changes to corundum and liquid.

Dumortierite has a practical advantage over the other three in refractories because of its higher  $\text{Al}_2\text{O}_3$  content.<sup>4</sup> In general, for determining its refractory nature, dumortierite may be assumed to be  $4\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$  and the thermal behavior may be determined from the alumina-silica diagram. At 1545° C. the liquid formed is about 4 per cent, which is considerably less than with andalusite, kyanite or sillimanite (14 per cent). The small amount of liquid formed from dumortierite as contrasted with the other three has a favorable effect on the load characteristics<sup>4</sup> of the calcined product.

Natural sillimanite can be heated for long periods at any temperature below 1545° C., without changing,<sup>2</sup> although all evidence indicates that the 1:1 compound has no stable existence at high temperatures. Above 1545° C. sillimanite breaks up into  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  and liquid. Sillimanite would be a desirable refractory if found in commercial quantities in accessible localities.

When any one of these four minerals changes to mullite, the new phases formed are of lower density than the original mineral. The expansion that results is negligible for andalusite and dumortierite, is slight for sillimanite and is considerable for kyanite.<sup>55</sup> Because of expansion on heating, kyanite may be used to advantage in combination with diaspore or bauxite in the preparation of refractories.<sup>13</sup> The expansion of kyanite will counteract the shrinkage of diaspore or bauxite.

Mullite is similar in optical properties to sillimanite, differing principally in refractive indices and axial angle.<sup>3</sup> The refractive indices of mullite may increase in value with  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  up to 4 per cent, when the values of  $\alpha$  and  $\gamma$  become virtually identical with normal sillimanite. In porcelain it usually forms fine needlelike crystals indistinguishable from sillimanite without optical determinations.

Since andalusite shows almost no volume change at dissociation, and kyanite shows great expansion and disintegration, treatment of the two minerals in ceramic practice differs greatly. Andalusite is usually molded into final form and fired directly<sup>40,41</sup> while kyanite is precalcined. The properties of the resulting porcelain may be as valuable in either case and the economic advantage of a more constant source of supply for kyanite may more than offset any advantage andalusite may have in requiring a single firing.

Kyanite, andalusite and sillimanite<sup>14</sup> have no definite temperature at which sharp decomposition occurs to form new phases; nor is there any considerable temperature below which the mineral remains unaffected for an indefinitely long period. In practice<sup>36</sup> kyanite breaks down at cone 12 (1350° C.) to mullite and glass, andalusite at cone 13 (1350° C.) and

sillimanite is not decomposed up to cone 15 (1435° C.). Kyanite and andalusite are decomposed with absorption of heat and yield mullite 87.64 per cent and silica 12.36 per cent.<sup>14</sup> Kyanite is more readily decomposed than andalusite.<sup>14,36</sup>

Measurements by X-ray, and crystal-structure determinations of andalusite, sillimanite and kyanite are fairly complete.<sup>6</sup> The unit cells and space groups of the natural forms of  $\text{Al}_2\text{SiO}_5$  are as shown in the table below.<sup>16,49,50,51,52,53,60</sup>

The unit cells for kyanite, sillimanite and andalusite each contain four molecules of the composition  $\text{Al}_2\text{SiO}_5$ . Mullite has a slight structural difference from sillimanite and shows a substitution of aluminum atoms for silicon atoms without any appreciable disturbance of the arrangement of the oxygen atoms. The unit cell for mullite has the dimensions:

$$a = 7.49 \text{ \AA.}, \quad b = 7.63 \text{ \AA.}, \quad c = 2.87 \text{ \AA.}$$

and the unit cell contains three-fourths of a molecule of  $\text{Al}_6\text{Si}_2\text{O}_{13}$ .

The electrical resistances of kyanite, sillimanite, andalusite and dumortierite have been determined by King<sup>26</sup> in ohms per cubic centimeter for fired material at various temperatures. Mullite derived from kyanite was found to have a better electrical resistance than mullite derived from any of the other members of the group.

TABLE 1.—*Structural Data*

	a, Å.	b, Å.	c, Å.	$\alpha$	$\beta$	$\gamma$
Kyanite. triclinc, $C_2'$ . . . . .	7 09	7 72	5 56	90° 51½'	101° 2'	105° 44½'
Sillimanite: orthorhombic, $V_h^{16}$ . . . .	7.43	7 58	5 74			
Andalusite: orthorhombic, $V_h^{12}$ . . . . .	7.76	7.90	5.56			

#### ORIGIN AND MODE OF OCCURRENCE

*Kyanite.*—Kyanite is a common mineral in areas of metamorphic rocks. It occurs in many places in the belt of crystalline rocks that traverses the states along the Atlantic seaboard of the United States. The mineral occurs: (1) in gneiss or schist in disseminated crystals, (2) as lenses in pegmatite dikes, (3) irregularly distributed through quartz veins or quartzitic masses, and (4) in residual clay formed by the weathering of kyanite gneiss. One of the most frequently associated minerals is quartz. The most important occurrences commercially are the deposits of disseminated crystals in gneiss or schist.

A disseminated deposit of considerable size is being worked at the extreme north end of Black Mountain Range, near Burnsville, N. C., in the Carolina gneiss (by Celo Mines Inc.). The deposit is cigar-shaped, 250 ft. wide by 2000 ft. long, and has been developed at one point with a vertical face of over 150 ft. exposed. It is reported to be steeply inclined (dip 77°) and has been assumed to have continuity with depth. The kyanite occurs in coarse platy crystals usually an inch or less in length but occasionally more than two inches long. The plates are about  $\frac{1}{16}$  in. thick and vary from  $\frac{1}{4}$  to  $\frac{1}{2}$  in. in width. Commonly associated with the kyanite are quartz, feldspar, biotite, numerous minute crystals of garnet, some muscovite and iron sulphides. Other minerals are tourmaline, beryl and staurolite.

The small scattered lenses found in pegmatites, the kyanite in the gold-quartz veins and the kyanite in the quartzitic masses are of little commercial importance. Residual and placer kyanites are being mined in Georgia<sup>39</sup> in small amounts.

Kyanite is thought to be formed under great pressure and high temperature in the crystalline rocks as a result of metamorphism of aluminous members of a stratified series.<sup>20</sup> Difference of opinion exists concerning the presence or absence of accompanying magmatic activity. The absence of intrusives in the immediate vicinity of certain deposits is cited as evidence against magmatic influence.<sup>20</sup> However, Bayley, Keith, Watson and Taber have given evidence<sup>21,22,48,57,58,1,44</sup> of igneous activity in the region and intrusives occur even in certain kyanite areas. It is difficult under such circumstances to separate unmodified dynamothermal metamorphism from dynamothermal metamorphism accompanied by the penetrating influence of confined igneous emanations. The association of beryl and tourmaline with the kyanite is an indication of pneumatolytic activity.

In India kyanite occurs in quartz-kyanite or kyanite schist and is associated with muscovite schist.<sup>10</sup> The kyanite deposits of the Bhandara district in India are of the schistose type and, although of little commercial value, are of interest because of the genetic relations observed. Both kyanite and sillimanite occur in the district but are mutually exclusive of each other. It is believed<sup>7</sup> that chlorite-muscovite schists yielded the rocks bearing kyanite and sillimanite as the result of pneumatolytic and hydrothermal metamorphism attendant upon the intrusion of adjacent granite and involving the separation of boric oxide or borates and occasionally vanadates derived from its residual magma liquor. Genetic relationships of minerals such as dumortierite, tourmaline, roscoelite mica and rutile, associated mica schist and prominent quartz veins support this theory of origin.

*Andalusite.*—Andalusite occurs in argillaceous schists, slates, gneisses and phyllites, and in granite pegmatites. It is commonly associated



TABLE 2.—*Physical and Optical Properties*

Mineral	Crystal System	Specific Gravity	Hardness	Cleavage	Optics <sup>a</sup>					
					$\alpha$	$\beta$	$\gamma$	Sign	Angle	Orientation
Andalusite ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ) . .	Ortho-rhombic	3.16-3.20	7-5	{110} good	1.634	1.639	1.643	Bx -	$2V = 85^\circ$ $r > v$ faint	$X = c$ $Z = a$
Sillimanite ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ) . .	Ortho-rhombic	3.23-3.24	6-7	{010} perfect	1.659	1.660	1.680	Bx +	$2V = 25^\circ-30^\circ$ $r > v$ perc.	$X = b$ $Z = c$
Kyanite ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ) . . .	Triclinic	3.56-3.67	Varies with direction	{100} perfect {010} good {001} parting	1.712	1.720	1.728	Bx -	$2V = 82^\circ$ $r > v$ slight	$X$ almost $\perp$ to {100} Ext. on {100} $Z \wedge c = -30^\circ \pm$
Dumortierite $8\text{Al}_2\text{O}_3 \cdot \text{B}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot \text{H}_2\text{O}$ . . .	Ortho-rhombic	3.26-3.36	7 4-7	{100} good	1.678	1.686	1.689	Bx -	$2V = 30^\circ-40^\circ$ $r < v$	$X \wedge c = 0^\circ \pm$ $Z = a$
Mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) . . . .	Ortho-rhombic	3.156	6-7	{010} perfect	1.638	1.642	1.653	Bx +	$2V = 45^\circ-50^\circ$ $r > v$ perc.	$X = b$ $Z = c$

<sup>a</sup> Optical data are given by Larsen and Berman,<sup>29</sup> Peck,<sup>23,27</sup> Schaller,<sup>43</sup> Wright,<sup>45</sup> Bowen,<sup>3</sup> and Greig.<sup>14</sup>

with tourmaline, corundum, topaz, garnet, pyrophyllite, quartz, biotite and muscovite. The andalusite at White Mountain, Mono County, California, occurs in irregular segregations in a lenticular quartz mass, with a narrow thickness of sericite schist on either side. The deposit appears to have been produced by a sequence of metamorphic proc-

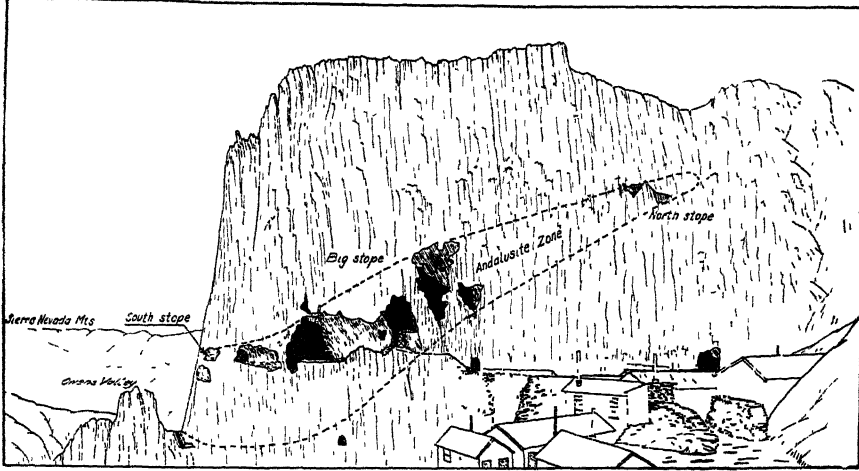


FIG. 2.—SKETCH ILLUSTRATING DISTRIBUTION OF ANDALUSITE IN QUARTZ MASS AT WHITE MOUNTAIN, CALIFORNIA. (*Econ. Geol.*, 1932, 27, 619.)

esses.<sup>11,23</sup> The original rock probably consisted of a series of trachytic flows with an intervening layer of aluminous volcanic material (or possibly an aluminous sediment). The andalusite was apparently

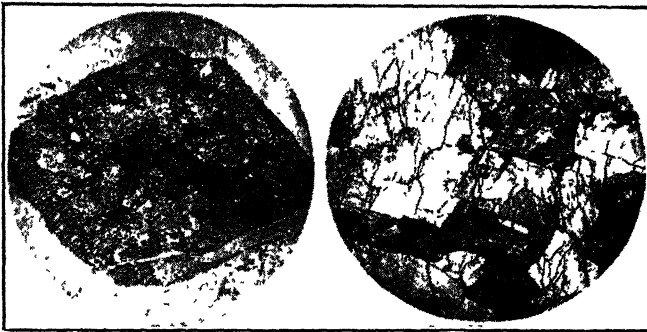


FIG. 3.

FIG. 4.

FIG. 3.—THIN SECTION OF ANDALUSITE CRYSTAL FROM WHITE MOUNTAIN, CALIFORNIA, SHOWING IRREGULAR QUARTZ AREAS SCATTERED THROUGH CRYSTAL.  $\times 10$ .

FIG. 4.—THIN SECTION OF ANDALUSITE FROM WHITE MOUNTAIN, CALIFORNIA. CROSSED NICOLS,  $\times 10$ .

produced by metamorphism of the original rock as one of the effects of an adjacent igneous intrusion.

The geologic features, wide distribution of titanium minerals throughout both the country rock and the andalusite mass, extensive tourmalini-

zation and inherited structures in the sericite schist, suggest pneumatolytic and later processes connected with an intrusive. It is thought that in the early stages of the formation of the deposit, metamorphism brought about the transfer of aluminous material from an aluminous rock to andalusite segregations bordering a quartz mass. The chief concentrations of andalusite have been found in the quartz not far from the mica schist. Tourmaline, topaz and diaspore, minerals indicative of igneous activity, were genetically related to the early mineralization. Abundant miarolitic cavities in the deposit also indicate gaseous influence. In brief, the andalusite deposit at White Mountain is probably due to pneumatolytic metamorphic action along the contact between a quartzitic mass and aluminous layer, resulting from igneous influence. Hydrothermal metamorphism at a later stage is indicated by extensive pyrophyllitization and sericitization.

Andalusite occurs near Hawthorne, Mineral County, Nevada, associated with corundum and diaspore, in a vertical veinlike deposit, 2 to 4 ft. thick, approximately 3000 ft. long and with an explored depth of 100 ft.\*

*Dumortierite*.—Dumortierite is found in pegmatite or quartz veins, which cut aluminous rocks such as cordierite or corundum gneiss or mica schist, and has been observed in kaolinized products of a micaceous granite. At Oreana, Nev.,<sup>19,37</sup> dumortierite occurs near the border and within a quartz mass formed by pneumatolytic invasions of a rhyolitic tuff.<sup>24</sup> The quartz mass is associated with a sericite schist in which occur irregular lenses of andalusite, partially or entirely altered to dumortierite. In massive character, size and association with a mica schist, it is similar to the quartz segregation containing andalusite at White Mountain, California.

The series of metamorphosed Triassic volcanics in which dumortierite occurs is not far distant from a granite intrusive.<sup>18</sup> It is likely that the emanations from this intrusive or hidden intrusives of a similar nature have forced their way upward through the Triassic series, bringing about crystallization of the andalusite and later dumortierite by metamorphism along the boundaries of the quartz mass adjoining the mica schist. The earlier phase was probably pneumatolytic when andalusite and earlier dumortierite were formed. Later, presumably hydrothermal metamorphism resulted in the formation of late dumortierite. Three generations of dumortierite were produced: (1) coarse euhedral blue dumortierite frequently found in crystals (probably early), (2) mottled lavender or pink masses (the bulk of the commercial ore) and (3) fibrous pink veins or isolated crystals.

*Sillimanite*.—Sillimanite occurs in gneisses, schists, slates and hornfels, and is probably produced at higher temperatures in nature than the

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\*Letter by Clifford Tillotson, Tillotson Clay Products Co., Los Angeles, Calif.

other minerals of the group, but under essentially similar conditions of metamorphism. It is found in separate crystals or in aggregations of slender minute prisms in radiating or diverse arrangement, often accompanied by garnet, cordierite, corundum, andalusite and rarely kyanite.<sup>7,39</sup> Important deposits occur in India<sup>10</sup> at Khasi Hills, Assam, and at Pipra, Rewa. At the latter place sillimanite is found in an area of sillimanite schist associated with corundum and surrounded by granitic gneiss. Both deposits are too inaccessible at present to be profitably mined.

*Mullite*.—Although common in refractories, mullite is rare in nature. In fact, it was only after identifying the aluminous silicate of refractories as  $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$  that Bowen and Greig found natural material of a similar composition in buchites described by Thomas.<sup>54</sup> The *buchites* are fused argillaceous sediments occurring rather frequently as inclusions in the Western Isles of Scotland<sup>14,45</sup> and were found to contain a product



FIG. 5.

FIG. 6.

FIG. 5.—THIN SECTION OF EUHEDRAL DUMORTIERITE CRYSTALS IN A QUARTZ SERICITE GROUNDMASS, OREANA, NEVADA.  $\times 10$ .

FIG. 6.—THIN SECTION OF SCATTERED CRYSTALS OF DUMORTIERITE IN QUARTZ SERICITE GROUNDMASS, OREANA, NEVADA.  $\times 10$ .

identical with the product of calcination in porcelain. This was called mullite after the Island of Mull.

#### DISTRIBUTION OF DEPOSITS

*Kyanite*.—Kyanite occurs in commercial quantities in the United States in North Carolina,<sup>47,30,22 48,12</sup> Virginia<sup>20,59,57</sup> and Georgia.<sup>39,1,44</sup> In addition to the large deposit now being worked near Burnsville, N. C., it is likely that other deposits capable of being worked exist in the mountainous region of the Black Mountain Range.<sup>13,30</sup> Noncommercial localities have been reported in Maryland,<sup>28</sup> Pennsylvania, Wyoming, Washington and California and in numerous places in the states already mentioned.<sup>20,21,39,48,58</sup> Kyanite is being mined in Imperial County, California, from a deposit covering about 15 acres at the base of the Chocolate Mountains, about three miles from Ogilby.\* Outside of

\* Letter by R. W. Ellison, Chief Chemist, Vitrefrac Corporation, Los Angeles, Cal.

the United States, kyanite occurs in many parts of the world in small amounts. The deposits of India are of chief importance commercially. Dunn<sup>10</sup> has estimated a minimum of 214,000 tons of kyanite at Lapsa Buru, India. Smaller and more or less inaccessible deposits exist at Ghagidih (20,000 tons), Badia-Bakra (10,000 tons) and Kanyluka (8,000 tons).

*Andalusite*.—The commercial exploitation of andalusite in the United States followed a lead suggested by Knopf,<sup>27</sup> who described andalusite from White Mountain, Mono County, Calif., in connection with studies conducted for the U. S. Geological Survey. Andalusite<sup>17,35</sup> is found in a number of separated occurrences on the north-western slope of White Mountain. It is also being mined in the vicinity of Hawthorne, Mineral County, Nev. It occurs in small amounts in the Black Hills of South Dakota.<sup>8</sup> The mineral has also been found in sands in western Transvaal, Africa.<sup>34</sup> Spaenhauer has described an interesting occurrence of andalusite and kyanite in Switzerland<sup>46</sup> and Corin has discussed the origin of andalusite containing manganese in Belgium.

*Dumortierite*.—Dumortierite is found in commercial quantities near Oreana, Nev.<sup>19,37</sup> Numerous noncommercial occurrences have been mentioned in the literature.<sup>19,27</sup>

*Sillimanite*.—Although this mineral occurs in sillimanite schist in a number of places in the eastern United States, the impure character of the material and frequent association with quartz in the form of fine needles renders commercial development difficult. The large deposits at Khasi Hills, Assam and at Pipra, Rewa, India, are as yet too inaccessible by rail or highway to be of commercial value. Dunn<sup>10</sup> has estimated 100,000 tons of available sillimanite at Pipra, which is about 105 miles by trail from the nearest railroad. Sillimanite has also been described associated with corundum in the norite of the Bushveld igneous complex, west Lydenburg, Africa.<sup>15</sup>

#### POLITICAL AND COMMERCIAL CONTROL

Commercial deposits of the aluminum-silicate minerals are largely in the hands of private companies. The andalusite mine at White Mountain, Calif. and dumortierite mine at Oreana, Nev., are controlled by Champion Sillimanite Inc. Andalusite is also mined near Hawthorne, Mineral County, Nev., by the Tillotson Clay Products Co., Los Angeles, Calif. Kyanite is mined at Ogilby, Calif. by the Vitrefrax Corporation of Los Angeles. McLanahan-Watkins Co., of Charlotte Courthouse, Va., operate a kyanite deposit. Celo Mines Inc. controls several kyanite deposits in North Carolina, particularly the large operation at the north end of Black Mountain. Chas. F. Taylor and Sons, Cincinnati, Ohio, import kyanite from India, about 100 miles west of Calcutta.<sup>41</sup> In

view of the comparatively small cost of these minerals in the form of raw material in proportion to the manufacturing cost necessary to produce high-grade porcelain, refractory companies prefer to own and operate their own deposits. Such ownership enables them to stabilize plant production on a uniform method of treatment based on the raw material available.

#### PRODUCTION AND CONSUMPTION

Because of the type of ownership, production figures are not regularly published. In the United States, production has been fairly steadily increasing since 1920. In the period 1922-1926, California produced 9394 tons<sup>38</sup> of aluminum silicate refractory minerals, largely andalusite. Production in recent years has probably been at about the same rate. During the past year and a half, dumortierite has been taken from stockpiles and mining has been discontinued. The McLanahan-Watkins Co. has been producing kyanite intermittently. The Vitrefrax Corporation estimates its production at about 800 tons of kyanite per year. Since the opening of the andalusite deposit at Hawthorne, Mineral County, Nev., on Sept., 1, 1935, five carloads have been shipped to the plant of the Tillotson Clay Products Co. in Los Angeles, for use in the manufacture of refractory products.\* Production of kyanite in India for 1933 was 4293 tons, by far the largest percentage being mined in the Singhbhum district.

Figures are not available for the amount of these materials used in the manufacture of the different porcelain bodies. The Champion Spark Plug Co., during a 10-year period, 1922-1932, using andalusite and dumortierite, produced 350,000,000 spark-plug cores.<sup>41</sup>

#### PROSPECTING, EXPLORATION AND MINING

The occurrence of minerals of the andalusite group is largely limited to areas of metamorphic activity or to detrital or transported material derived from metamorphic rocks. Pegmatites and associated intrusives yield some andalusite and kyanite, but on the whole have not been commercially productive. Prospecting and exploration are largely confined to areas of surface exposure or near surface occurrence. Metamorphism is usually intense and results in rather a wide distribution of low-grade material. High-grade concentrations are comparatively rare.

Mining methods are for the most part extremely simple. Open cuts or short tunnels are used to penetrate the deposits. In the California and Nevada occurrences, the material is mined selectively and hand-sorted, the final selection of material being judged by frequent tests of specific gravity. In the piedmont belt the kyanite is mined in open

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\* Letter by Clifford Tillotson, Tillotson Clay Products Co., Los Angeles, Calif.

pits, and, wherever possible, in the residual deposits, separated from the enclosing material by washing. At Cleo Mines, Burnsville, N. C., a series of 20-ft. benches has been blasted parallel to the strike of the deposit, and, after secondary blasting, the ore is collected by drag scrapers on a loading platform.

#### PREPARATION FOR MARKET, TESTS AND SPECIFICATIONS

*Preparation.*—The andalusite of the Champion Sillimanite Co., in California, requires selective mining and hand-sorting before shipment to Detroit. At White Mountain, Calif., the andalusite must be carried by packmules down a steep trail,  $4\frac{1}{2}$  miles, and then transported by truck to a storage platform along the narrow-gauge railroad, 3 miles from the foot of the trail. At the siding in California and at the factory in Detroit, the material is spread horizontally in long, parallel strips, making a pile containing 2000 tons. For uniform grade the material is withdrawn at right angles to the original spreading direction. Dumortierite receives similar treatment.

At the plant the andalusite and dumortierite ore is crushed, passed over a magnetic separator, sized and distributed to storage bins. Bricks of dumortierite alone gradually swell and those of andalusite sag, a condition that can be remedied by a proportionate mixing of these components with each other or with other clay materials.<sup>38</sup> In making porcelain the components are mixed with water, reground to 320 mesh, the excess water removed in a filter press, the "cakes" stored for 10 days in an aging cellar to develop plasticity and then formed into "blocks" from which spark plugs may be made. Spark-plug cores are burned in a continuous kiln 300 ft. long at 3700° F., the process requiring four days for the cores to pass through the kiln.

The kyanite in the deposits of North Carolina, Georgia and Virginia is irregularly distributed, ranging from 2 to 80 per cent in volume in relation to the enclosing rock. The McLanahan-Watkins Co., at Charlotte Courthouse, Va., uses a simple wet gravity process for concentrating the ore, which occurs in a decomposed rock and residual clay 40 to 50 ft. deep. Celo Mines Inc., Burnsville, N. C., uses a dry process; that is, the material is crushed and then fed to a machine with special hammers, which disintegrates without grinding the individual crystals. Screen concentration eliminates about one-third of the original ore with little loss of the kyanite, which is caught on the coarser mesh screens. An induction type magnetic separator removes, in addition to the abraded iron, the sulphides, garnet, biotite and a part of the muscovite, and a pneumatic concentrating table removes the quartz and small amount of feldspar and muscovite.\*

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\* Paper by V. L. Mattson, presented before the American Ceramic Society, April 1, 1936.

The Vitrefrax Corporation, Ogilby, Calif., calcines the kyanite ore in a rotary kiln, which reduces the iron for removal by a magnetic separator and converts the alpha quartz to beta quartz. Quenching the calcine in water reconverts the beta quartz to alpha quartz, causing expansion. Later contraction produces a fine sand, which can be separated from the coarser kyanite calcine by slightly inclined shaking screens, the relatively pure kyanite fibers remaining on the screen.<sup>41</sup>

*Tests and Specifications.*—The minerals of the andalusite group are seldom found free from mineral associates. Some, such as muscovite, diaspore, pyrophyllite and sericite, do not detract from the value of the ore if present in moderate amounts. Quartz, however, which is one of the most persistent associates, must be kept as low as possible. Pyrite, magnetite and limonite, if present in more than traces, must be removed.

The most convenient commercial test is specific gravity. Commercial andalusite, for example, should have a specific gravity of at least 3.00 to be considered high grade. The Kraus Jolly balance or some other type of improved balance<sup>25</sup> for rapid weighing may be used.

#### MARKETING, USES AND PRICES

Synthetic mullite is important, for refractories for spark-plug porcelain, glass tanks in the glass-blowing industry, saggars, furnace-lining mufflers and parts of furnaces subjected to high temperatures.

The dumortierite from the mine at Oreana, Nev., is used exclusively for the manufacture of spark plugs by the Champion Spark Plug Co. The company markets both dumortierite and andalusite refractories as "Champion" sillimanite. The Vitrefrax Corporation produces refractory products under the trade names "Argon" and "Durex." The Corhart Refractories Co., in Louisville, Ky., manufactures a vitreous mullite refractory, using diaspore and kaolin, very suitable for glass-tank blocks. The kyanite and mullite produced by the Celo mines is being used for the manufacture of refractories, particularly for glasshouse refractories and refractory cements.

The cost of the raw material is the limiting factor in a more widespread use of the minerals of this group. When introduced, kyanite sold for \$100 a ton, but this was soon reduced to \$40 a ton and more recently the price has been slowly but steadily decreasing.<sup>61</sup> In the period 1922–1926, California produced 9394 tons, mostly andalusite, valued at \$301,790 or about \$32.13 per ton.<sup>38</sup> In 1932 kyanite was quoted in lump or concentrated form at from \$25 to \$80 per ton, according to purity, grain size and whether calcined or raw.<sup>59</sup> At the end of 1934 the Celo Mines Inc. quoted \$18 a short ton for 70 to 80 per cent kyanite, grading up to \$25 a ton for 90 per cent kyanite. An additional charge of \$15 a ton was made for calcining.<sup>61</sup>

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\* Letter by V. L. Mattson, Celo Mines Inc., Burnsville, N. C.



In 1928 the Singhbhum district in India produced 2241 tons of kyanite valued at £2509, a little over a pound per ton.<sup>38</sup> In 1933 the output was 4283 tons at £5220, a slight increase in price.<sup>61</sup> These prices are f.o.b. Amda, a station on the Bengal-Nagpur railroad. The price of sillimanite in England in 1929 was £11 per ton, but had been £14 per ton shortly before.<sup>10</sup>

High alumina-silica refractories were quoted in 1932 as follows: Corhart fused mullite, standard shapes \$200 per ton, P.B. sillimanite refractories (using kyanite from India, Chas. Taylor company) 9-in. brick \$500 a thousand, Babcock and Wilcox calcined kaolin fire brick \$260 a thousand, 9-in. diaspore brick (70 per cent alumina) \$145 a thousand.<sup>59</sup>

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## CHAPTER III

### ASBESTOS

By J. G. ROSS,\* MEMBER A.I.M.E., AND GEORGE F. JENKINS†

**A**LTHOUGH asbestos deposits have been actively exploited for the past 70 years, it is only within the last 30 years that asbestos has become one of the important commodities of commerce, having required the needs of modern industry and science to develop its many uses. In early Greek literature, Pliny uses the word "asbestos" in reference to a fibrous mineral; Roman literature refers to "amiantus," which—as also the "asbestos" of the Greeks—was employed in the weaving of cremation cloth, and for lamp wicks. Because of its appearance, it was probably first considered an organic substance. Marco Polo, in writing of his travels in the thirteenth century through Siberia, in the district of Chinchutalus subject to the Great Kublai Khan, relates that: "A substance is likewise found of the nature of the salamander, for when woven into cloth, and thrown into the fire, it remains incombustible." He continues that this "fossil substance" was prepared, spun, and woven into cloth, and "in order to render the texture white, they put it in the fire and suffer it to remain there about an hour, when they draw it out uninjured by the flame and become as white as snow. And so again whenever they become dirty, they are bleached by being put into the fire."

Although the discovery of asbestos is attributed to the Romans, who mined it in a small way in the Alps, interest in it apparently lapsed and little mention is made of it in the literature of the Middle Ages. About 1710 to 1720, discoveries were made in the Ural Mountains, and 40 years later, during the reign of Peter I, a factory for the manufacture of asbestos articles was established in Russia. But the known uses were so few, and the demand so limited, that the industry subsequently disappeared. Later, interest in asbestos was revived in Europe, and technical investigation begun as to its properties and utility. From the year 1860, the search for deposits became widespread, and real interest in their development began to appear. The first modern attempt at exploitation was made by a London syndicate in the Aosta Valley of the Italian Alps, and production started in 1865. About the same time, asbestos was discovered between the villages of St. Joseph and St. Francis in Quebec. At

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the exhibition in London in 1862, a specimen of fine silky-fibered asbestos from the above locality was exhibited. No deposits were developed here however, and it was not until 1877 that the important discoveries at Thetford and Coleraine—in the same belt of serpentine rocks—were made. Production started the next year.

Prices at first were comparatively low, even for the best grades of fiber, but gradually increased after 1885. From that year the industry gathered impetus and expanded rapidly. At this time, systematic development began in Russia and, a few years later, in Africa. By 1905, while Canada was the chief source of supply, Russia and South Africa were producing, and Rhodesia was added to the list in 1908. For many years Canada led in production by a wide margin, but in 1934 was surpassed in gross value of asbestos produced by Russia.

#### COMPOSITION AND PHYSICAL PROPERTIES

The word “asbestos” as used today is a term embracing the fibrous varieties of a number of minerals. Some of these, in their more common forms, are found as constituents of basic igneous rocks, and of major importance in the composition of certain metamorphic rocks. They are included in two mineralogical subdivisions: the amphibole group and one of the hydrous silicates of magnesium, serpentine. While these minerals are common, those possessing the necessary physical properties to be of commercial importance are not abundant; and, regardless of chemical composition, when crystallized with highly developed prismatic cleavage so that the mineral may be easily separated into fibers or filaments, it may be classified under the generalized commercial term, “asbestos.”

There are sometimes considerable differences in the physical properties of different species of asbestos minerals, and often marked variations between specimens of the same mineral, even if obtained from near-by deposits or the same deposit. The mineral's value as asbestos is determined by certain characteristics that make it particularly fitted for some specific use or uses by industry. If it separates readily into long silky fibers of good tensile strength, it is generally suitable for spinning into yarn; if the fibers are less pliable but still strong, it will be more suited for use as a binder or reinforcing agent such as is necessary in the manufacture of asbestos-cement shingles or magnesia pipe covering. Short, easily separable fibers are used in the manufacture of asbestos paper, while coarser or more brittle types are unsuited for this purpose. Any of the several minerals used as asbestos are, to a degree, fireproof and acid-resistant, but some species may better withstand high temperatures or attack by acids than others; some are more hygroscopic than others—that is, have more tendency to absorb moisture from the atmosphere. So it may be realized there are several factors to be investigated in considering

the commercial value of any given kind or form of asbestos mineral. Such characteristics as hardness and specific gravity are relatively unimportant; but color and possible associated impurities do sometimes have a bearing on the value.

The cleavage of an asbestos mineral is responsible for the quality of fine silkiness of the fibers. Several of these minerals are characterized by an unusually high development of the prismatic cleavage, which permits separation along innumerable planes to a remarkable degree of fineness. Individual filaments have been measured to as small as  $1/25,000$  in. in diameter, and these, in turn, might be divided into still finer shreds.

The minerals that may occur in sufficiently fibrous form to be generally recognized as asbestos are: (1) amphiboles (sometimes called hornblende), under which are classified anthophyllite, tremolite, actinolite, crocidolite, amosite; (2) serpentine (or chrysotile).

#### AMPHIBOLE

*Anthophyllite* is a silicate of, essentially, magnesium and iron, having the chemical formula  $(\text{MgFe})\text{SiO}_3$ , often with some replacement by aluminum and calcium. It occurs as mass fiber, the crystals being unoriented and interlacing; sometimes, in a radial arrangement. Commonly it is grayish white; occasionally, shades of green or blue. The fiber of this species is usually brittle, although sometimes it has considerable strength and toughness. It cannot, however, be considered of spinning quality, and its chief use is for insulation, in cement or plaster, or in the manufacture of paint. A certain short-fibered variety known as microasbestos—probably anthophyllite—is used with cement or asphalt in road building.

*Tremolite* is a calcium-magnesium silicate  $(\text{CaMg}_3(\text{SiO}_3)_4)$ . It occurs in masses of white to dark gray columnar or bladelike crystals, which generally are brittle, and are used principally for wall insulation or certain cemented products. This mineral has been found, however, occurring as fibers of sufficient length and flexibility to be of spinning grade. As it has particularly good acid-resisting quality, it is especially suited to the manufacture of filter pads, used in filtering fruit juices and acids.

*Actinolite* is similar in appearance to tremolite, but contains iron replacing some of the magnesium  $(\text{Ca}(\text{Mg.Fe})_3(\text{SiO}_3)_4)$ . It sometimes occurs as bright green or grayish green fibrous masses. The filaments are too brittle for spinning purposes, but are highly resistant to heat and acids; it is otherwise employed for the same uses as tremolite.

*Crocidolite*, because of its bluish gray or lavender-blue color, is commonly called "blue" asbestos. Occasionally it occurs in yellowish or rusty brown shades. This species is a complex, soda-iron silicate, the chemical formula of which is  $\text{NaFe}(\text{SiO}_3)_2 \cdot \text{FeSiO}_3$ . It is commercially the most important variety of amphibole asbestos, and occurs as cross-

fiber veins\* consisting of filaments of good length and flexibility, and can be spun into coarse yarn. It does not withstand high temperatures as well as the other asbestiform amphiboles, but is quite resistant to the action of chemicals. The purposes for which crocidolite is suitable are numerous and are included in many of the items under Uses.

*Amosite* may be described as an iron-rich amphibole, without the sodium molecule of crocidolite and, except for its higher iron content, similar in composition to anthophyllite. The percentage of water of crystallization is generally higher in crocidolite and amosite than in the other minerals of this group, analyses showing between 2.55 and 4.15 in the former, and 1.05 to 4.7 in the latter. It is commonly of an ash gray color, or more rarely white or pale brown, and occurs in cross-fiber veins several inches thick, thereby having exceptional fiber length. The fibers are pliable but of rather coarse texture, and it is not used extensively in spinning. It has been found satisfactory as a binder, or reinforcing, for heat-insulating blocks such as magnesia pipe covering.

### SERPENTINE

Serpentine is a hydrous silicate of magnesium having the chemical formula  $H_4Mg_3Si_2O_9$ . It is—as, in fact, are the asbestiform varieties of amphibole—a secondary mineral, being formed by the alteration of other minerals. It occurs in several forms: the dense, massive variety is common serpentine; that composed of bladed, columnar, or coarsely fibrous crystals is called picrolite; while the highly fibrous variety is known as chrysotile.

*Chrysotile*.—Of all the minerals used industrially as asbestos, chrysotile constitutes by far the largest percentage of the world's supply. Veins of cross-fiber chrysotile are usually of various shades of green, although sometimes light yellow or cream color and, in some deposits, of a light brownish shade. When the crystals are pulled apart, or “opened,” the color of the fibrous mass is usually white—as is true of most kinds of asbestos, except crocidolite, which retains its bluish hue. The better grades of chrysotile are characterized by their extreme fineness of fiber, or “silkeness,” which, coupled with high tensile strength and sufficient length, make it particularly valued for spinning. The percentage of water of crystallization, which may have some bearing on the fiber flexibility of all varieties, is highest in chrysotile, being commonly from 10 to 13 per cent. This factor of constitution affects the heat-resisting qualities adversely, chrysotile being weakened at lower temperatures than amphibole asbestos, with the exception of crocidolite.

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\* By common usage, the word “veins” is accepted, although pertaining here to tabular bodies of dimensions which, in the more widely used sense, would be called veinlets or stringers.

Magnetite is a mineral that is sometimes closely associated with the structure of a chrysotile vein. When it is present the fiber is less suited for electrical insulation. Some deposits are iron-free, and the fiber from these is particularly prized for electrical purposes. Generally speaking, throughout the wide range of uses for which asbestos is adapted—from short grades of dustlike consistency, to the longest grades—chrysotile supplies the major portion for nearly all of the major uses.

#### ORIGIN AND MODE OF OCCURRENCE

An asbestos deposit seldom contains more than one asbestos mineral. Two or more physical forms of the same mineral may occur closely associated, and one variety may be found in rocks of widely divergent origin and character. The main feature in common to all deposits is some degree of metamorphism, this action, in many cases having been severe. Dependent upon the type of metamorphism and the kind of rock affected, a generalized classification may be arranged:

1. Serpentine type (developing chrysotile asbestos). Alteration to serpentine of:
  - a. Basic igneous rocks such as dunite and peridotite.
  - b. Sedimentary rocks such as dolomite and limestone.
2. Amphibole type. Amphibole asbestos developed in:
  - a. Impure shales and slaty quartzites such as the siliceous ferruginous slates of the so-called banded iron-stone formation.
  - b. Basic igneous rocks.
  - c. Highly altered rocks of gneissoid or schistose character, of igneous or sedimentary origin; gneiss, gneissoid schist, or crystalline schist of high magnesia content.

Asbestos is commonly found in three forms, according to the mode of occurrence:

*Cross-fiber*, when the fibers are arranged normal to the vein walls; the fiber length is thereby limited to the vein width and is often less, due to a break, or parting, in the vein;

*Slip-fiber*, when the fibers lie in the same plane as the vein or slippage plane upon which it is formed;

*Mass-fiber*, when formed as an aggregate of interlaced unoriented fibers, or as stellate groups of radiating needles.

Some 90 per cent of the world's asbestos supply is derived from deposits of serpentine asbestos in dunite and peridotite. These are intrusive rocks composed essentially of olivine, peridotite differing from dunite in that it contains an appreciable percentage of pyroxene. Large masses of these rocks have undergone various degrees of serpentinization. This alteration was probably accomplished by solutions emanating from the same magmatic source as the basic intrusives themselves or, more directly, from solutions accompanying later acidic intrusions such as granite or aplite. By these the magnesian silicate, olivine, was altered



to the hydrous magnesian silicate, serpentine. In places, the entire mass was permeated sufficiently to produce a widespread and high degree of serpentinization. Chrysotile veinlets were formed along fractures, where conditions were favorable.

Certain portions of the formation, commonly forming roughly ellipsoidal bodies, and probably resulting from segregation during solidification of the original rock, were especially favorable to vein development. In these bodies, which are often fairly extensive, a network of veins may be found constituting up to 15 or 20 per cent of the volume. These chrysotile veins range from a fraction of an inch to one, two, or more inches wide, and are generally short, or continuous for no more than 50 to 100 ft. They may intersect each other at various angles or may lie in closely spaced parallel planes, giving the appearance of stratification. This latter arrangement is called "ribbon structure."

Aside from these higher grade bodies, the serpentine rock often contains also a low percentage of cross-fiber, and, in some cases, up to 5 or 10 per cent or more of slip-fiber. Slip-fiber is more abundant in the softer, more highly serpentinized zones, although frequently it occurs on faulted or slickensided surfaces in the more massive rock. Apparently it was formed with the aid of dynamic action; by the movement producing a fault, or the slight displacement on a slippage plane; or, in a mass which has yielded to pressure by "flowing" in the solid state, to the extent of developing a scalelike structure. In the latter case, slip-fiber is found in the slickensided serpentine covering the surface of each small scale.

Examples of the foregoing types of chrysotile deposits are numerous in the serpentine belt that extends from northern Vermont northeasterly through the Eastern Townships of Quebec. The bulk of production is from deposits worked primarily for the cross-fiber content, although slip-fiber occurs to some extent in most of the ore bodies. A few mines are operated on ore which contains little or no cross-fiber.

The asbestos deposits being mined in the Shabani district of Southern Rhodesia, and those of the Bajenova district in the Urals, Russia, are similar in mode of occurrence to the cross-fiber bodies of Quebec. In Cyprus, small veins of chrysotile are found in the periphery of a large peridotite plug.

Chrysotile veins occur in limestone and dolomite within bands and lenses of serpentine, or in the outside shell surrounding nodules or larger ellipsoidal bodies of massive serpentine; they form as successive layers of asbestos and serpentine enveloping the serpentine core. The more extensive deposits of this type are those in bands or flat-lying lenses of serpentine; the veins occurring as layers paralleling the bedding of the limestone. In Arizona, deposits are found in the portions of a dolomitic limestone associated with diabase sills. Solutions from the diabase have serpentinized these parts of the limestone, with the accompanying

development of asbestos veins. Similar occurrences have been worked in the Carolina district of the Transvaal and in Minusinsk, Siberia.

Of the amphibole varieties, crocidolite and amosite are the only ones commonly having the cross-fiber habit. Both minerals occur as interbedded veins in banded ironstone, which is a silicified slate formation containing iron. Commercially important deposits of crocidolite are found in Cape of Good Hope, South Africa. Here the ironstone formation is extensive and occurrences are widely distributed throughout the belt. The fiber ranges from less than  $\frac{1}{2}$  in. to 2 in. and longer. The proportion of long fiber to short is greater than in deposits of chrysotile, such as those of Canada, Russia and Rhodesia, although the chrysotile mined in the Barberton district of the Transvaal is said to be even longer than the crocidolite from its neighboring state, Cape of Good Hope.

Amosite is mined in northeastern Transvaal, where it also occurs in banded ironstone formation. There are no intrusive rocks associated with the deposits in this formation.<sup>26</sup> Its occurrence is very similar to that of crocidolite, and the two minerals are closely related. Crocidolite is found in the western part of the amosite belt, both varieties being recovered from some of the workings. Amosite fiber is unusually long, averaging about 6 in., and ranging upwards to 10 or 12 inches.

The other varieties of asbestiform amphibole, anthophyllite, actinolite and tremolite, occur largely as mass-fiber or, in some cases, as slip-fiber. In deposits such as those of Sall Mountain, Georgia, anthophyllite occupies pockets and lenses in a gneissic rock of probably igneous origin. The ore bodies occur as fibrous masses, which may contain up to 90 or 95 per cent commercial fiber. Near the surface, where subjected to weathering, the fiber is softer and more suitable for use than below the weathered zone.<sup>2</sup> Anthophyllite is also found as slip-fiber occupying shear zones, and rarely as cross-fiber. It is generally harsh and rather brittle, but there is a hard, dense type, which may be broken down into a soft, fluffy mass. It is rarely found of sufficient length and silkiness to be of spinning quality.

In Italy the early production consisted of tremolite. The fibers are often long but frequently too coarse to be valued for spinning.

#### DISTRIBUTION OF DEPOSITS

The known asbestos resources of the United States are of limited extent. Exploitation and development have been retarded by mining and transportation difficulties in some localities, but there is nowhere indication of extensive deposits such as are found in several other countries.

The serpentine belt that extends from Quebec into northern Vermont, has, in Vermont, yielded comparatively small tonnages of the medium

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<sup>27</sup> References are at the end of the paper.

and short grades of chrysotile asbestos. The fiber is of good quality, but no extensive deposits have been developed. The limestone deposits of Gila County, Arizona, have been worked, producing a very good grade of chrysotile. However, mining and transportation costs have discouraged development; mills have been built, but the mines are active only when high prices prevail. Other occurrences of chrysotile have been explored and, in some cases, small amounts produced in California, Montana, Wyoming, North Carolina and Virginia.

The largest tonnages of amphibole asbestos produced in the United States have come from the Sall Mountain district of Georgia. Here, owing to the fact that the best material is within the zone of weathering, the deposits are shallow, and reserves apparently have neared

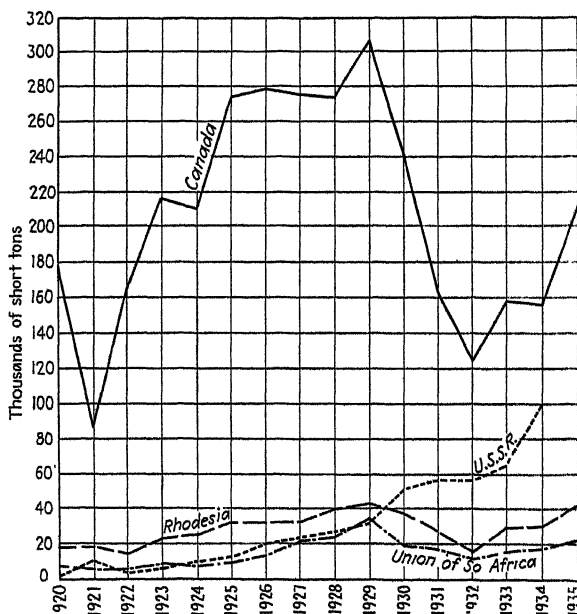


FIG. 1.—ASBESTOS PRODUCTION BY COUNTRIES.

depletion. Small-scale operations, working the shear zones in a gneissoid schist near Pylesville, Md., yield anthophyllite suitable for acid filter. Some development work has been done on amphibole occurrences in Montana, North Carolina, Virginia, Idaho, California, Oregon and Washington, but no potentially large supplies have been found.

Outside of the United States a number of extensive asbestos deposits have been proved. Chrysotile occurrences of the type found in Quebec, Southern Rhodesia and the Bajenova district of Russia probably extend to considerable depth, which would add to the large reserves already developed. Those of the Barberton district, in the Transvaal, containing chrysotile have not been intensively exploited though from 8,000 to 17,000

tons per year has been produced. Difficult transportation, and the fact that the beds are banded and dip into the ground make extensive underground development necessary to block out large tonnages. Some of the chrysotile deposits of the Carolina district, of the same province, occur in serpentinized basic igneous rock, and are of the same type of occurrence as those mentioned above, while others are in dolomite and similar to those of Arizona, and of Minusinsk, Siberia. No large reserves have been disclosed. Cyprus has a supply of short-fibered chrysotile.

The only commercially important occurrences of crocidolite and amosite are those of South Africa, where the fiber-bearing belts of Cape of Good Hope and the Transvaal cover a large area and have been worked

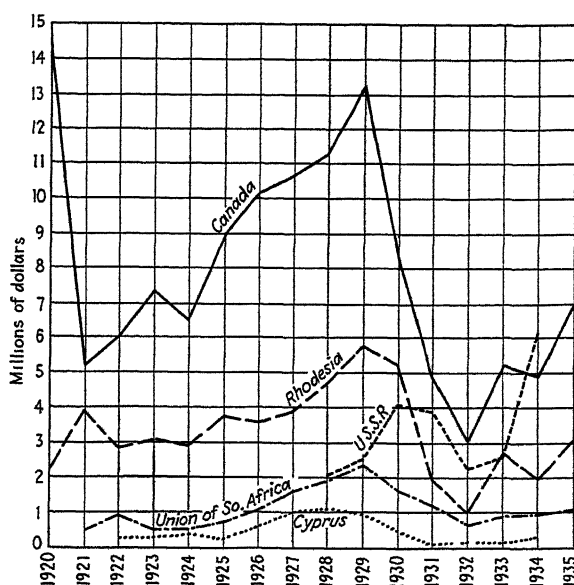


FIG. 2.—VALUE OF ASBESTOS PRODUCTION.

as small operations in many places throughout the extensive ironstone formation. Many other countries have known deposits, most commonly of chrysotile asbestos, which are small, low grade, or developed to only a limited extent. Among these are Italy, Finland, Czechoslovakia, India, China, Western Australia, New South Wales, New Zealand, Madagascar, Turkey and Venezuela.

#### POLITICAL AND COMMERCIAL CONTROL

Aside from those of Russia, all of the large producing deposits of asbestos lie within the British Empire. English capital controls the mines of Rhodesia, South Africa, and Canada, although in Canada it shares this with large interests of the United States and Canada. English

own the productive area of Cyprus. The Russian mines and resources are, of course, exclusively owned by the State.

The United States is by far the greatest consumer of asbestos, and imports the larger part of its requirements from Canada, buying some 90 per cent by quantity, or 80 per cent by value, from this source. This normally accounts for over 30 per cent of the Canadian production. For many years, England, France, Germany and Belgium imported the major portion of their supply from Canada, but the African mines now furnish much of England's large manufacturing needs, while Germany, Belgium, Italy and central European states are supplied, in varying proportions, from Canada, Russia, and, to a lesser extent, from Rhodesia, while France, Spain and Portugal draw supplies from Canada and Rhodesia.

The rapidly expanding production of asbestos in Russia has disturbed world markets but to a much smaller degree than might have been anticipated. Russia's program calls for increased manufacture and home consumption of asbestos products, with the result that much of the raw product is not produced for export, and therefore is not competitive with that from other countries. It now ranks second only to the United States in the quantity of asbestos used.

Japan produces a negligible percentage of the fiber it uses. It imports largely from Canada, and a little from Russia and Africa.

For the shorter, lower priced grades, Canada holds an advantageous position. Much of this material is used in the United States, and because freight charges constitute a larger proportion of the delivered cost than for the higher priced fibers, its proximity to these markets makes it difficult for other countries to compete in these grades.

Asbestos is an important commodity of international trade. Aside from Russia, none of the important producing countries are substantial consumers of their raw product, nor are any of the large consuming countries—other than Russia—able to supply more than a small part of their own needs.

#### PROSPECTING AND MINING

Asbestos deposits are resistant to weathering, and often are less affected by erosion than the surrounding rocks. For this reason outcrops frequently occupy knolls or ridges, and are more easily seen than deposits of less stable minerals. The kind of formation in which the more common types of deposits occur is easily recognized, and prospecting may be thus confined within definite boundaries. When an outcrop is found, or uncovered by trenching or stripping, the grade of ore exposed can be closely estimated by visual inspection, particularly when it has been broken into by drilling and blasting. Asbestos veins, where long exposed to weathering, are often discolored as a result of the oxidation of impurities. The fibers generally remain in place between the vein walls, except for the upper few inches that have been exposed.

Where overburden is present, the usual methods of trenching and stripping are used to reach the solid rock. Further exploration may be carried on by diamond or calyx drilling; the calyx, or shot drill, has the advantage of giving a larger core and, therefore, a better sample for examination, but is not practical for deep drilling. The majority of deposits, however, are developed by test pits, which are simply continued and enlarged into quarries when results are satisfactory.

Exploration by tunneling or other methods of underground excavation, or by diamond-drilling from these workings, is sometimes advisable, depending upon the position of the area to be explored.

The percentage of asbestos content, when derived by other means than visual examination and estimate, is determined by mill recovery from large bulk samples, or by laboratory methods simulating the operation of milling.

Many of the standard open-cast and underground mining methods are used in the extraction of asbestos ore. A few innovations, particularly in handling the rock of open-cast workings, are peculiar to the industry. Underground mining systems are similar to some of those used in metal mining, with occasional variations to suit special conditions.

Open-pit workings have a wide range of size and shape, depending upon the area and depth of the deposit, and the extent to which it has been exploited. In these the rock is broken in such a manner as to allow it to fall to the quarry bottom or the faces are advanced in a series of benches. The ore in the majority of deposits may be classed as soft or medium hard, to drill and break. Air drills of the common hammer type are in general use, mounted on tripods for drilling flat breast holes, and often for steep bench holes as well.

Holes with this equipment range up to 18 ft. in depth. Deeper drilling, to 40 ft. or more, is accomplished by using a heavy drill with an A-frame and slide arrangement; or drill carriages are used, either type of equipment allowing steel changes at intervals up to 10 ft. For block-holing and other short holes, various sizes of jackhammers are used, in accordance with standard practice. Wet drilling is uncommon, as water mixed with asbestos fiber forms a spongy mass, which is difficult to clear from the holes. The dust, at least from most Canadian asbestos ores, has no evident deleterious effect on the workmen's health.

The broken rock is handled in various ways. Hand-loading methods are common even in some of the larger operations in Canada, and are used almost entirely in Russia and Africa. These methods persist partly because they permit more thorough sorting of waste rock. Ore is shoveled or barred-down into open-mouthed steel boxes, or shoveled directly into cars or other vehicles. Where boxes are used, they are handled and dumped into cars by means of boom derricks, steam cranes or overhead cableways. If cableways are used the boxes are hoisted directly to sur-

face, obviating other hoisting facilities. Elaborate systems of overhead cableways were developed in the Thetford mines district of Quebec, but are little used now.

Some pits are relatively shallow to the area covered, and ore trains are run directly to the working faces. One large mine uses a spiral track layout following benches from which the mining is carried on. Standard-gauge steam or electric locomotives, and ore cars are run to the pit bottom over several miles of track. In other mines, where the relative depth precludes the use of locomotives to reach the pit bottom, hoisting is used: on an incline, through incline tunnel, or by shaft. In any case, where large-capacity ore cars are brought to the working faces, the usual method of loading is by power shovel, or by cranes handling steel boxes. The boxes are loaded by hand, the reason being that more effective separation of waste rock from ore can be achieved in the quarry by hand-loading.

Extraction by the glory-hole system is used by some mines, and may be effected from any type of underground opening. Raises are put up from an established level to surface, where the ore is broken down by funneling out the top of the raises, and is drawn off through these into cars.

Various types of underground mining methods have been used in asbestos mines. For flat-lying deposits, under barren formation, likeing many of those in sedimentary rocks, flat stope or a system of room-and-pillar mining is necessary. For steeper or deep deposits, shrinkage stoping, and, at least once, sublevel stoping has been used. In the most compact serpentine rock, stope widths can be safely carried at 40 ft. or more. A recent development in one mine of the Thetford district is the application of a form of block caving. In this mine the method is briefly as follows: Opening is by vertical shaft, and one main haulage level is established at a depth of 500 ft. from surface. The latter is served by electric locomotive and 5-ton cars. The overlying ore is divided into blocks 160 ft. square in plan. A block is developed by four grizzly drifts running through it at 40-ft. intervals, and at an elevation of 45 ft. above the haulage level. These drifts are timbered with 6-in. H-beams, and finished flush with concrete. (Steel timbering is used throughout the mine in preference to wood, as it is desirable to prevent wood chips from contaminating the fiber.) Each pair of grizzly drifts is served by one drift on the haulage level below, with which it is connected by ore-pass raises. Grizzlies of heavy steel rails with 16-in. opening between rails are constructed at the top of each raise. From each grizzly—these being spaced at 20-ft. intervals along the drifts—two finger raises are put up, one on each side of the drift. At 20 ft. higher, the block is completely undercut by driving drifts and blasting out the intervening pillars. The finger raises are funneled out to permit large pieces of ore to be reached and broken up at the grizzlies, when actual extraction is begun. In order to weaken the boundaries of the block, raises are put up at each corner and

connected by drifts. These are called boundary cutoff drifts, and are spaced according to the character of the rock. It is desirable that the material cave within vertical walls, and these boundary drifts form a line of weakness for controlling the break. All development work having been completed and the pillars of the undercut level shot out, the ore breaks down from its own weight. As the broken ore supporting the back is drawn off through the finger raises, by working it through the grizzlies, more caves to take its place. This action continues through to surface. Where it is necessary to protect property in the vicinity of the mine workings, the walls of the caved block are retained by backfilling with mill tailings (these are of the consistency of fine gravel), which are dumped in from surface, and follow the broken ore as a plug as it is drawn off.

#### PREPARATION FOR MARKET

In the early days of production, asbestos was supplied to the market entirely in the crude form, and the only portion of the asbestos content of an ore body that was recovered consisted of the larger veins, which could be conveniently picked up as lumps and roughly dressed by hand methods. In 1888, a few years after production had started in the Quebec fields, mechanical means began to be developed to aid in preparing cleaner, more uniform crude, and to recover some of the shorter fiber then being rejected. Difficulties were encountered, but by 1896 sufficient progress had been made with milling to produce important tonnages of fiberized asbestos. The demand for these fibers increased, and milling practice has improved and expanded, until at present there are many mills of daily capacities from 1000 to 2000 tons, and individual plants capable of treating 4000 to 6000 tons of ore daily. Nevertheless, hand dressing persists in the recovery and preliminary preparation of crudes. It is an effective means of recovering the long fiber without subjecting it to the rougher treatment of machines, with the accompanying effect of sacrificing fiber length. A much larger proportion of the asbestos is recovered by hand in some fields than in others.

The Canadian mines are highly mechanized, and Russia, in recent years, has followed the methods developed here. The Rhodesian asbestos mines employ a larger percentage of hand labor for fiber recovery, but milling has become an important part of the process. Hand methods predominate in the South African fields, but even there, mills with elaborate flowsheets have been built. The one mill in operation in Vermont closely follows Quebec practice; those of Georgia and Arizona are small. The following brief description of Canadian practice includes the essentials of most of the methods used elsewhere.

*Cobbing.*—When the ore is broken in mining, many of the larger veins are partly loosened from their walls. Where accessible, these pieces



of vein material are picked up by hand immediately. Some lumps retain portions of the wall rock, which are freed by the blow of a hammer. Further recovery of the larger pieces of cross-fiber veins (slip-fiber is not generally of sufficient value to be handled in this way) is effected sometimes on picking belts or tables before or after the primary crusher. The material thus obtained is further treated in a department separate from milling; and the whole operation is known as cobbing. The rough crude is dried and screened, after which each piece that has any adhering rock or contains impurities (massive serpentine and magnetic iron are the more common impurities) is pounded by hand with a special hammer, to break up and free the undesirable portion. Lumps that are off-color, or otherwise below standard in quality, are rejected. The best material, of a fiber length of  $\frac{3}{4}$  to 1 in. and above, is classified as No. 1 Crude; the remainder, down to  $\frac{1}{2}$  or  $\frac{3}{8}$  in., is graded as No. 2 Crude. After cobbing, each grade is further cleaned on screens. Except where special preparation is desired, the crude is now ready for bagging and shipment. In some places, run-of-mine crude or other classes are produced. The cobbing and screening rejects are sent to the mill, or treated separately as special grades.

*Milling.*—The release of asbestos from the rock and from accompanying impurities is effected in milling by following much the same principle as in cobbing. The fibrous structure is strong and tough, while the gangue is brittle. Crushing, impact, and grinding break up the rock and associated minerals; while the asbestos is simply separated along its cleavage planes and converted into a mass of fibers in a fluffed condition. A certain amount of fiber is cut or broken but, to minimize this, the machines used are carefully selected or specially designed. Separation of the released material cannot depend on differences of gangue and the mineral to be won, such as chemical composition or specific gravity, as is the basis of treatment of most ores. The principle by which asbestos is removed from the crushed ore is that the volume of the mineral is increased by any action that tends to separate the individual crystals, thus converting it into a fluffed condition. Because of this, the ore is adaptable to screening to remove the fines produced in crushing, and the fiber to being removed by a current of liquid or air. All existing mills use air as the medium of separation, and the ore must be dry for effective milling.

The earlier mills effected separation by blowing the crushed or ground ore into long chambers; the heavier rock particles falling first and the more highly fluffed fiber being carried to the farther end. Later practice developed the use of suction hoods, placed at the end of shaking screens, operating on the principle of a vacuum cleaner. Large fans produce the necessary vacuum, and hoods, with a narrow opening extending across the lower end of a screen, distribute the air currents, allowing the fiber to

be swept from the rock and aspirated through large sheet-iron pipes. Enlarged sections of the circuit, in the form of collectors, recover the fiber. Modern mills, in order to recover the dust and very light, short fiber not caught by the collectors, discharge the air from the fans into large dust sheds, where this fine material may settle.

There is considerable variation in the equipment of one mill as compared to another, but the flowsheet of each aims to have machinery that most effectively frees and separates the asbestos from the ore with the least injury to the fibers, and adequate to recover all of the salable fiber and by-product grades. Some ore bodies contain principally short fiber, and others a large percentage of long; either characteristic making for simplified milling, particularly the latter when remoteness from the market makes it uneconomical to ship the cheaper grades. Canadian mines, because many of the ore bodies contain appreciable amounts of both long and short fiber, and because the relative proximity to markets makes the production of low-priced grades possible, have developed milling practice to a high degree, and produce by far the greatest range of fiber grades. As a rough guide to the process and the kind of equipment used, a generalized flowsheet, showing alternate machines of different mills, is given herewith.

### *Generalized Flowsheet*

#### PRIMARY CRUSHING

Jaw or gyratory crusher

Picking belt or tables (recovery of crude and, in some cases, rejection of waste)

Grizzly

Jaw crusher, gyratory, or rolls

Grizzly

Gyratory or swing-hammer crusher

} or cone crusher

#### DRYING (Material size about 2 inches)

Cylindrical rotary drier or vertical stack drier

#### BINS

Storage to provide uniform mill feed, curing, and surge

Capacities range up to 25,000 tons

#### MILL (Secondary crushing and primary separation)

Screens and suction

Cone crusher, hammer mill, gyratory or rolls

Screens and suction

Torrey cyclone, hammer mill, cone crusher, rolls, or jumbos

Screens and suction

Jumbos, cyclone, or ball mill (seldom)

Screens and suction

Sometimes—one or more repetitions of the last stage, generally using jumbos. This may be in the form of a separate tailing unit

Tailings conveyed to dump

The fiber from the preliminary separation contains sand and scales of rock, etc. This must be eliminated by further screening, and lifting the fiber by suction. The asbestos recovered at the head of the mill is the

longest, and is generally finished separately. It is graded on flat screens or, more commonly, in stationary trommels called graders, which take out the correct amount of the shorter portion to arrive at the desired grade, besides aiding materially in the cleaning process. The shorter fiber is further cleaned on screens, and, with similar fiber recovered in the primary separation, is sent to other graders to produce the second longest grade. In some mills, the operation is repeated to make three, four, or more grades. Additional screening and, sometimes, grading stages are used after the preliminary grading in order to produce cleaner fiber, and arrive at the correct test. At this point graders may prepare the fiber in a more opened, or fluffed, condition, if desired.

The material collected in the dust shed is returned to the mill, where short fiber is extracted, and the dust used in producing various extremely short grades called "floats."

The finished fibers fall into bins from which they are bagged (100 or 125 lb.) by hand, or with the aid of bagging machines.

While a limited number of fiber grades can be made in a mill at one time, by equipment adjustment and minor alterations other combinations of grades can be readily produced. An explanation of grading and testing will be given on another page.

### MACHINERY

Certain of the equipment peculiar to asbestos milling requires a brief description:

*Hammer Mill.*—The hammer mill consists of a modified cylindrical housing, one size commonly used being 54 in. long and about 22 in. at the shortest radius (inside dimensions). It is lined with manganese-steel plates. A horizontal central shaft is fitted with disks and spacers, and, near the periphery of these disks, six equally spaced smaller shafts are passed. On these shafts are hung steel arms or "hammers" at  $2\frac{1}{2}$ -in. intervals throughout their length. The assembly is rotated at high speed, causing the hammers to swing outward. The rock, which is fed from the top of the housing, is struck by the revolving hammers and thrown against the steel lining, being crushed by impact. It is discharged through an opening that runs the length of the bottom of the housing. The opening is either wide enough to allow free discharge or is constricted by grids or bars.

A similar machine has been fitted with air suction, and used as the principal equipment in the recovery of anthophyllite asbestos in Georgia.<sup>2</sup>

*Cyclones.*—Two principal types of cyclones are used. The Torrey cyclone consists of a vertical, cylindrical shell enclosing a central upright shaft. Ore, fed at the top, falls onto a small table revolving rapidly with the shaft. Centrifugal force throws the material to the outside of the table, from which it is guided by vanes and caused to impinge on deeply

grooved castings lining this part of the shell. Falling from there into a conical hopper, the rock is guided to a second table, from which it is again thrown and further crushed by impact.

The other cyclone is a smaller machine. Two, heavy, three-bladed propeller-like beaters are arranged close together on separate horizontal shafts. In the Laurie machine these are revolved in opposite directions, while in another make, the Pharo cyclone, they rotate in the same direction. Feed comes in at one end; falling between the beaters (which are face to face), it is broken by impact on the blades and against the sides of the bowl-shaped receptacle, and ground under the beaters. Discharge is upward, and is induced by the material being thrown and blown out by the high speed of revolution.

*The Jumbo.*—This machine has largely replaced the Laurie and Pharo cyclones. It consists of a horizontal cylinder, 6 or 8 ft. long, assembled with cast-iron ends and corrugated cast-iron liners. A number of arms, fitted with heavy, detachable tips, are clamped on a horizontal shaft, and revolve at a moderate rate. The feed, entering the top at one end, is subjected to a rubbing and grinding action as it is moved the length of the machine by the beater tips, which have beveled faces of a pitch sufficient to impart forward motion to the feed at the desired rate. Discharge is through a hole in the bottom at the end opposite the feed.

*Driers.*—Rotary driers are 40 to 60 ft. long and 4 to 6 ft. in diameter, and inclined at a flat angle. Hot air from a firebox passes from one end to the other of this cylinder, and is brought more closely in contact with the material being dried, by large angle irons running lengthwise on the inside of the shell, which cause the rock to be lifted and cascaded as the machine slowly revolves.

The vertical type of drier is built as a square stack 50 ft. high and 7 by 7 ft. in cross section. The crushed rock falls through a rising current of hot air and gases coming directly from a firebox built on one side at the bottom. The falling material is retarded by grid bars placed at regular intervals throughout the height of the stack.

*Screens.*—Various sizes and types of screens are employed. Shaking screens actuated by eccentrics are the most common. The speed, length of stroke and slope used vary widely. Usual sizes are from 3 to 5 ft. wide and from 8 to 12 ft. or more long. Perforated steel plate is more generally employed than wire mesh, particularly on rock screens. Perforated plate with holes up to  $\frac{3}{8}$  in., and larger for scalping screens, is used at the head end of the mill. Smaller openings down to  $\frac{1}{32}$  in. are required for very fine material. Different sizes of wire screening, ranging in mesh from 3 to 24 to the inch, are frequently used on fiber-cleaning screens. Vibrating screens find limited application in some mills.

*Suctions.*—The hood through which fiber is lifted from the lower end of a screen is adjustable, and has approximately the same cross-sectional

area at the mouth as the pipe into which it converges. The width of opening is generally from 2 to 5 in., and piping leading to the collector is from 12 to 20 in. in diameter. One fan generally furnishes two or more suctions, which may lead into one collector. For a suction pipe 15 in. in diameter, from 10 to 15 hp. at the fan is required to produce the desired flow of air and lifting power.

*Graders.*—A grader is a cylinder of perforated steel plate through which passes a central shaft. Arms are clamped to the shaft, and detachable tips bolted to the end of the arms. A popular size is 8 ft. long and 28 in. in diameter. The arms and tips are twisted at a slight angle, which, with the speed of revolution of the shaft, governs the rate at which the fiber is moved through the machine. The speed is usually between 400 and 900 r.p.m. Material enters one end; undersize is forced through the perforations of the plate by the tips (there is a small clearance between tips and shell), and oversize is discharged at the opposite end.

#### TESTS AND SPECIFICATIONS

The grades in which fiber is classified are controlled within stated limits by means of a standard testing machine developed in the Quebec field, and almost universally accepted as the unit of measure by which milled asbestos is sold. This machine consists of a nest of four rectangular screens held in wooden frames, placed on a table that is actuated by a shaft and eccentric. The bottom box serves as a pan, and the screen of each of the three superimposed boxes is of successively larger mesh. The machine, boxes and size of wire and mesh are made to exact specifications. The screen of the top box has  $\frac{1}{2}$ -in. openings between wires, that of the second is 4 mesh, and of the third, 10 mesh. Sixteen ounces of the fiber to be tested is placed on the upper screen, and a cover clamped over the top, securely holding the nest of boxes. The machine is started, runs at 300 r.p.m. for two minutes, and stops automatically. The boxes are removed; the fiber remaining in each is weighed and recorded to the nearest tenth of an ounce. With this machine, fiber can be tested ranging from nearly all minus 10 mesh to that of sufficient length to be largely retained on the top screen. Ro-Tap machines, using a nest of small sieves, are also employed, assisting in the control of production of short fibers.

Until recent years, each company designated its own grades. This caused confusion and inconvenience to the buyer. In Canada, the condition has been remedied by the Quebec Asbestos Producers' Association, which, in conjunction with the Government, has agreed upon uniform design of testing equipment, a standard control in the form of a Government testing machine, and standard markings for the various grades. Fibers are divided into groups, and each group subdivided into grades, which are designated by letter prefixed by the group number.

- Group No. 1. Crude No. 1 ( $\frac{3}{4}$ -in. staple and longer).
- Group No. 2. Crude No. 2 ( $\frac{3}{8}$ -in. staple up to  $\frac{3}{4}$ -in.). Crude run of mine (unsorted crudes). Crudes, sundry (crudes other than above specified).
- Group No. 3. Textile and spinning fibers (testing 0-8-6-2 and over).
- Group No. 4. Shingle fibers (testing below 0-8-6-2 and including 0-1.5-9.5-5.0).
- Group No. 5. Paper fibers (testing below 0-1.5-9 5-5.0 and including 0-0-8-8).
- Group No. 6. Waste, stucco, or plaster fibers (testing below 0-0-8-8 and over 0-0-5-11).
- Group No. 7. Refuse or shorts (testing 0-0-5-11 and below).
- Group No. 8. Sand.
- Group No. 9. Gravel and stone.

As examples of the manner in which these groups are subdivided into grades; a 3K fiber is one of the spinning and textile group, having a minimum test of 4-7-4-1; a 5R fiber is one of the paper-fiber group, and has a minimum test of 0-0-10-6.

Some plants produce two or more types of the same grade, characterized chiefly by the degree to which the fiber is fluffed, or opened.

#### MARKETING, USES AND PRICE

Most of the raw asbestos produced is sold to manufacturers of asbestos products. Agencies and representative firms handle part of the business, and in some cases act as jobbers, but most of the fiber is shipped direct from mine to consumer. Samples are usually submitted to the customer, and sales made on guaranteed test and quality. Formerly, large and long-term contracts were common, but now contracts are made to terminate at the end of the current year. The supplies of raw asbestos are in strong hands, as well as much of the manufacturing, and close cooperation between the two is usually found. Mutual understanding is particularly desirable in the asbestos industry, since, to supply the manufacturer's needs to the best advantage—particularly in the milled fiber field—the producer should be familiar with conditions in order to prepare the material to suit the particular use and process for which it is required. Several of the large manufacturing companies control their own asbestos mines.

More of the fiber, in recent years, is sold in a condition ready for use in the plant without the necessity of further opening and cleaning action. Some mill fibers and all crudes, however, are supplied in a less opened state, and the manufacturer subjects it to the final stages of fluffing and elimination of waste.

The uses for asbestos are many and diverse, and space will not permit enumeration except of a summary nature. Much of the longer fiber is spun into yarn, which is woven into such fabrics as brake linings, tape and cloth. The cloth may be used for various purposes, including brake linings, gaskets, theater curtains, insulating mats, awnings, aprons and gloves. Explorers that have descended into the craters of active volcanoes have worn asbestos clothing.

A portion of the longer grades is used as wick and rope packings, filling for mattress insulation, and substantial quantities in magnesia-block insulation, consisting of 85 per cent magnesia and 15 per cent asbestos.

The fact that it is the only fibrous mineral binder accounts for many uses where asbestos aids in holding together and reinforcing cement products, plasters, asphalt, and molded articles of the cold-molded and bitumen type, and those made with synthetic resins of the phenolformaldehyde type.

Shingles, siding, and other forms of asbestos lumber are made from medium-length grades of fiber by mixing with cement. Asbestos tile

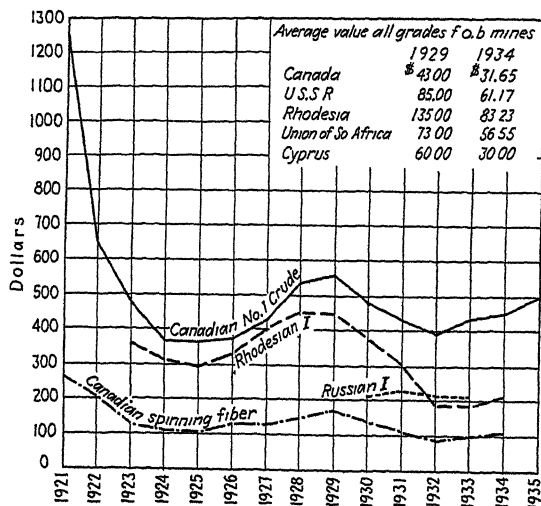


FIG. 3.—YEARLY AVERAGE VALUE PER SHORT TON OF CANADIAN NO. 1 CRUDE AND SPINNING FIBER.

Sales prices f.o.b. mines. Rhodesian I and Russian I. Quotations c.i.f. European ports.

pipng and conduit are also of this class. These grades also are processed with rubber in the manufacture of sheet packing and associated products including certain kinds of brake lining.

Paper stock, as the name applies, is used largely in the manufacture of asbestos paper. Much of this is made into air-cell and other types of boiler and pipe covering. The development of molded brake lining and clutch facing, which are made from heavy paper of the millboard variety impregnated with certain compounds, has created additional demand for these fibers at the expense of the spinning grades.

Asbestos cement, widely used for boiler insulation, is made from the waste or plaster grades. These and refuse or shorts are constituents of cold-molded and synthetic resin products, various kinds of flooring, as filler in paints and greases, and of other compositions.

The many uses for which asbestos is so peculiarly adapted make it invaluable to industry. No single substance combines the variety of

properties that this does; it withstands fire and insulates against heat and sound; it is light in weight, and can be made into pliable fabrics; it resists soil corrosion and the attack of vermin. Uses are ever expanding and undoubtedly it will continue to play a prominent part in the development of transportation, building, and other industries.

Prices of the various fiber grades cover a considerable range. Floats and other very short grades sell down to \$10 and less per ton; cement stocks from \$20 to \$25; paper stock from \$30 to \$40; shingle fibers range from \$45 to \$65; and spinning fibers are generally something over \$100 per ton. Prices for Canadian crudes, for the last 10 years, have averaged about \$450 per ton for No. 1 and something less than \$250 for No. 2. Early in 1921 quotations for No. 1 crude reached a high of more than \$3000 and three years later had a quoted low of \$300 per ton. The prices for shorter fibers fluctuated less violently, but shingle stock sold for over \$100 in 1921.

The graph of Fig. 3 shows average values based on yearly sales for Canadian No. 1 crude and spinning fiber, while the Rhodesian I and Russian I are plotted from average price quotations, according to statistics published by the Quebec Bureau of Mines.

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## CHAPTER IV

### BARIUM MINERALS

By W. M. WEIGEL,\* MEMBER A.I.M.E.

ONLY two minerals of barium are produced commercially—barite ( $\text{BaSO}_4$ ) and witherite ( $\text{BaCO}_3$ ). Barite is also called “baryta,” “barytes” and “heavy spar,” and locally in Missouri, “tiff.” Witherite sometimes is also called “heavy spar.” More often it is simply called barium carbonate.

Pure barite is white opaque to transparent. Impurities sometimes give pale shades of yellow, green, blue, brown, red or gray-black to black. Barite crystallizes in the orthorhombic system. Twinning is common. The fracture is uneven and the mineral is brittle. Hardness varies from 2.5 to 3.5 and specific gravity from 4.3 to 4.6. Different localities are known to produce either “hard” or “soft” barite. The soft variety is more desirable for grinding. The streak is white. The luster is pearly to vitreous or sometimes stony. Theoretically barite contains 65.7 per cent BaO and 34.3 per cent  $\text{SO}_3$ .

Witherite is white, gray or yellow. It is transparent to translucent to opaque. The streak is white. It crystallizes in the orthorhombic system. It is brittle with uneven fracture. The hardness varies from 3 to 3.75 and the specific gravity from 4.29 to 4.35. It effervesces with cold dilute hydrochloric acid as distinguished from barite. Its theoretical composition is BaO, 77.7 per cent;  $\text{CO}_2$ , 22.3 per cent.

#### ORIGIN AND MODE OF OCCURRENCE

Barite deposits, according to their mode of occurrence, may be loosely grouped under three classes:

1. Those in which barite is a gangue or accessory mineral in metaliferous deposits.
2. Veins, replacements or breccia filling in sandstones, shales, limestones or crystalline rocks. Replacements sometimes occur as bedded deposits due to the complete replacement of sedimentary beds.
3. Residual deposits in clays, derived from the weathering of barium-bearing rocks. The most typical of these are the residual deposits resulting from the weathering of Cambrian or Ordovician dolomites.

Deposits of the first class have but little economic importance as far as the barite is concerned, it being usually difficult or impossible economically to separate a barite product of standard commercial grade.

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Deposits of the second class constitute an important economic group and are the source of production in some localities. They are also the source from which the third class is derived. The third group is of the greatest economic importance in the United States, as it includes the principal producing fields of Missouri, Georgia and Tennessee. Deposits of the second class are found in rocks of practically all ages from Archean<sup>11</sup> to Mesozoic.<sup>5</sup> Most of the commercial deposits of this class are in limestone or dolomite, but a few are in the crystalline rocks.

*Fissure Veins, Replacements and Breccias.*—In the Hot Springs and Kings Mountain area of North Carolina the veins are irregular in shape, varying from a few inches to 18 ft. wide.<sup>11</sup> The enclosing rocks are granites of Archean age, feldspathic quartzites of Cambrian age and fine-grained, white, siliceous sericite schist of Algonkian age. Barite veins in the Appalachian Valley of Virginia occur in Beekmantown (Lower Ordovician) limestones and dolomites. Calcite, quartz and pyrite are associated minerals. Some fluorite is nearly always present. In the Piedmont province of Virginia the veins are in pre-Cambrian crystalline rocks. The associated minerals tourmaline, hornblende, biotite, epidote and chlorite are thought to be distinctive of these deposits.<sup>5</sup> Barite veins in the central part of Kentucky are simple fissure or fault fractures in Ordovician limestone. The associated minerals are calcite, fluorite, galena and sphalerite. Barite occurs with fluorite in nearly vertical veins in Mississippian limestone in the western part of the state.<sup>9</sup> Strong veins of barite occur in Carboniferous rocks west of Boulder, Colo. Near El Portal, Mariposa County, Calif., the barite occurs as lenses, averaging 14 ft. with a maximum of 35 ft., in a vein of varying width. Both barite and witherite are found in this deposit.<sup>2</sup> At Lake Ainslie, Cape Breton, Canada, barite associated with calcite and fluorite occurs in veins in pre-Cambrian felsite. Near Five Islands, Nova Scotia, fissure veins and brecciated zones are found in Devonian slates and quartzites.

Replacement deposits are sometimes associated with veins, especially in sedimentary rocks. They usually show gradational boundaries in true replacements and sometimes merge into filled breccias where some of the brecciated fragments have been partly replaced with barite. Edmundson<sup>5</sup> says that in Virginia they are in the form of lenticular bodies, sometimes up to 20 ft. in diameter and are best developed in limestone and marble.

A replacement of shale by barite occurs in central Arkansas.<sup>1</sup> The deposit has replaced shale beds at the base of the Stanley formation of Mississippian age. It lies on the north flank of a steep synclinal valley, the west end of which contacts the igneous rocks of Magnet Cove, about 1½ miles distant. The known thickness is 44 ft. normal to the bedding plane. The bedding planes have been preserved in the replacement.

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<sup>11</sup> References are at the end of the paper.

Replacement deposits of barite in Carboniferous limestone occur in Belgium. Near Meggen, Westphalia, a bituminous barite is supposed to have replaced portions of a bedded Devonian pyrite, which in turn grades into limestone.<sup>9</sup>

Some breccia deposits resemble veins and are often associated with them. They are the result of the filling of the voids in the original brecciated zone rather than a clear-cut single fissure or fault, as with a vein deposit. Few of them are of economic importance in themselves but the barite in some of the residual deposits may be the result of the weathering of barite-filled breccias.

*Residual Deposits.*—By far the greatest production of barite in the United States is from residual deposits formed by the weathering and disintegration of rocks in which the barite was originally deposited in the form of veins or breccias. Probably few of them are the result of the weathering of replacements.

The residual deposits of southeastern Missouri are found in the clay mantle covering the underlying dolomites. These dolomites originally contained the barite in the form of veins or breccia fillings and the weathering of the rock left the less soluble barite in the residual clays. The dolomites are of Cambrian age. Deposits on the Potosi formation of the Cambrian are the most important but the overlying Eminence formation accounts for some. The deposits consist of loose fragments of barite, with chert, drusy quartz and boulders of dolomite and sandstone embedded in the red clay. There is often a concentration of the larger fragments near the bottom or even on bedrock. The clay mantle varies from 0 to 30 ft. thick. Areas of 100 acres or more may be ore-bearing but there is usually a concentration of the barite in runs or "leads" 10 to 20 ft. wide and several hundred feet long. A typical section is as follows:<sup>14</sup>

Surface soil.....	1 ft. to 1 ft. 6 in.
Chert, gravel and clay .....	1 ft. 6 in. to 2 ft.
Clay, barite, chert and drusy quartz, barite in small fragments, not over 2 in.....	2 ft. 6 in. to 4 ft.
Practically barren, dark red clay.....	3 ft. to 4 ft.
Red clay, large chert and dolomite boulders and large masses of barite, sometimes in almost continuous sheets 4 to 6 in. thick and several square feet horizontal extent	1 ft. to 2 ft. 6 in.
Red clay, practically barren, depth to bedrock not exposed.	

The barite is pure white on a fresh fracture, although the surfaces and cleavage planes are usually coated with a thin film or stain of limonite. The larger lumps often have deep cavities on their surfaces lined with a layer of hard limonite  $\frac{1}{32}$  to  $\frac{1}{16}$  in. thick. Massive galena is sometimes associated with the barite.

In the central Missouri district the residual deposits are often found in clays on top of the weathered surface of the Gasconade formation, the

top member of the Cambrian, but sometimes on the Roubidoux, the bottom of the Ordovician, or near the contact of these two. The mode of occurrence is much the same as in southeastern Missouri except that the deposits are deeper and of very restricted horizontal extent. The barite is a harder variety and is less stained and coated with limonite. Drusy quartz is absent. These deposits are probably more often the remains of filled breccia deposits than of fissure veins.

The residual deposits of Virginia have been the source of most of the production from that state. In the Piedmont region barite is found in red to black clay, the remains of the weathering of limestone and mica schist of Cambrian and pre-Cambrian age. In some places ocher and umber of commercial grade is associated with the barite, as are also manganese oxides. In the Valley region the enclosing clays were derived from the Knox dolomite.

In the residual deposits near Cartersville, Ga., the barite occurs as pebbles, nodules and masses embedded in the residual clays derived from the weathering of the Weisner quartzite and shaly limestone of the Beaver formation overlying it. These are the two lower formations of the Cambrian. The barite originally was deposited in fractures and cavities in these rocks. Some of it was possibly a replacement of part of the limestone. In the upper part of the deposits, on the slopes of the hills, concentrations of iron ore rest directly on the quartzite. The barite occurs below this on the slope with a rather sharp line of division between. The barite and its enclosing clay is usually underlain by remnants of the lower beds of the limestone, which have been completely weathered under the iron ore.

The residual deposits near Sweetwater, eastern Tennessee, are in clays averaging 10 to 20 ft. thick but occasionally reaching a thickness of 60 ft. These clays cover the Knox dolomite of Cambro-Ordovician age. The barite is of both the hard crystalline and soft varieties. It is iron-stained and sometimes coated with a hard layer of limonite.

*Origin.*—Clarke states<sup>3</sup> that most of the occurrences of barite and witherite indicate minerals of aqueous origin. This refers to deposits of barite in place in veins, breccias and replacements. It is generally accepted as a fact that the barite in residual clay deposits is the accumulation of the barite in bedrock deposits caused by the weathering of the enclosing rocks.

There are two schools of thought on the source or origin of the barium in the mineralizing solutions from which it was deposited in the various types of deposits found in place. One holds that most of the deposits were derived from descending meteoric waters and the other that they were deposited from magmatic or ascending waters. In either case the original source of the barium was in the igneous or crystalline rocks. If the meteoric waters theory is adhered to, the weathering of these rocks

permitted the barium compounds to be deposited along with the sedimentaries resulting from the remains of the weathering or erosion of the original rock. From this position the barium was picked up in solution by descending or circulating meteoric waters and deposited or precipitated from these mineralized solutions in veins, breccias or as replacements in adjacent or underlying formations where conditions were favorable. The chemistry of the process is explainable. In some deposits the points in favor of meteoric origin are sound and difficult to refute. For example, there may be no igneous rocks in near association with the deposit. Some of the associated minerals are known to occur in deposits formed by meteoric waters, which are rarely found in deposits of magmatic origin. There is often no alteration of wall rocks such as would be expected if they were subjected to hydrothermal action. If the deposits are due to magmatic waters these would have taken up their barium compounds from deep-seated igneous rocks or from intrusives or other rocks affording a source of the barium. These mineralized solutions, then, in ascending deposited the barite where conditions of rock structure, temperature and chemical action were favorable. In some deposits the evidence in favor of magmatic origin seems quite conclusive, such as silicification of the wall rocks, association of minerals of definite hydrothermal origin, close association of igneous or intrusive rocks, lack of sufficient barium in surrounding or overlying formations to afford the amount of barium present in the deposit, and lack of evidence of solution or leaching of adjacent rocks.

The various deposits of the world show such a diversity of occurrence that examples can be cited in which the evidence in favor of each theory is strong. It is probable that barite deposits have been formed by both processes.

#### DISTRIBUTION OF DEPOSITS

Deposits of barite occur in all parts of the world. This wide distribution, coupled with the fact that low-priced substitutes for many of its uses are available, reacts to keep it a comparatively low-priced commodity. For this reason development has taken place only where a market is readily available, mining costs are low and low-cost transportation is near at hand.

*United States.*—Deposits in the United States that have produced recently are principally as follows. Near Cartersville, Ga., is one of the most important districts. The North Carolina deposits are near Hot Springs and Kings Mountain. The Kings Mountain district extends southwestward into South Carolina and at one time was developed at Kings Creek, Cherokee County. Barite occurs in Virginia in 19 counties. The two most important areas are in the southwestern and central southern part of the state. In Kentucky there are many vein deposits in the

central part of the state near Lexington, but production has never been large. There are also deposits in the western part of the state. The Tennessee deposits, near Sweetwater, are of very considerable commercial importance. Alabama was a producer up to a few years ago. In Missouri, the state with the greatest production, the principal area is in Washington County, in the southeastern part of the state, with deposits of lesser importance in the central area southwest of Jefferson City. Deposits near Canon City and Boulder, Colo., have accounted for small production at various times. Producing veins in California are in Mariposa, Nevada and Shasta Counties. The mine at El Portal, Mariposa County, is the only producer of witherite in the United States. It also produces barite. A deposit on the west coast of Prince of Wales Island, Alaska, was at one time a small producer. Small amounts of barite have been produced at intervals in Esmeralda and Mineral Counties, Nevada, Sierra and Dona Ana Counties, New Mexico, Lafayette County, Wisconsin, and Blaine County, Idaho. Deposits have also been noted in Connecticut. A large undeveloped deposit is near Magnet Cove, Ark. The Llano district, in central Texas, is reported to have possible economic deposits.

*Foreign.*—Many foreign countries have deposits of barite, but Germany, Italy, Canada, Great Britain and Russia are the principal ones with commercial production entering into world trade. Spain produces some barite, some of which is exported. Germany's most important deposits are in the Thuringian Forest, the Black Forest, the Odenwald, Hesse and the Bavarian Palatinate. In Italy the principal producing districts are Carrara, Iglosias, Milano and Trento. Canada's principal commercial deposits are on Lake Ainslee, Nova Scotia. Other deposits, which have accounted for a small production, are in Ontario.<sup>10</sup>

England has been the chief producer of witherite. The deposits are in Northumberland County, in the north of England. Deposits of barite are developed in Northumberland, Durham, Cumberland, Westmoreland and Yorkshire Counties. Deposits of barite are noted in other parts of England. Deposits in Scotland are commercial producers.

Russia has entered the field of producers within the past few years. Deposits are reported at numerous localities. High-grade concentrates are produced at Salair, U. S. S. R.<sup>4</sup> Undeveloped deposits are reported in western Siberia, the larger of which is near Askyskom. Others are in the region of Morozoc Ulus, and at Chapsordag and Taptan-Tursy in the Hakassia district. There are producing mines in Georgia and Azerbaidzhan. New deposits are reported in the Urak, Siberia, Karelia, the North Caucasus and Central Asia.<sup>6</sup>

#### POLITICAL AND COMMERCIAL CONTROL

Most of the principal consuming countries of the world have sufficient supplies of barite for their own use. There is, however, some interna-

tional flow of crude or ground barite. This movement is based more on final cost at the point of consumption than on origin, source of supply or national boundaries. Factors entering into this are cost of mining and dressing or concentrating, transportation, tariffs, and grade of product. For example, the United States has ample reserves and potential production to take care of all its needs, yet a considerable tonnage is imported for consumption along the Atlantic seaboard because the delivered price is lower than that of domestic barite from the principal fields in Georgia and Missouri, owing to lower European production costs and low ocean rates compared with high American labor costs and higher rail rates.

The United States and Germany are the two largest producers. In 1931 and 1932 the United States production exceeded that of Germany. In 1933 and 1934 German production exceeded that of the United States. During the past few years Russia has become a factor in world trade. In 1913 Russia was an importer, while now it exports considerable amounts. The first exportation to the United States was in 1931. Outside of Russia's entrance into the world markets, there has been little change in the political control within the past few years, except some fluctuation in imports and exports of the principal countries. German exportation to the United States has decreased greatly and that from Spain has increased sharply. These fluctuations have been controlled probably more by trade balances and monetary exchange than by actual supply and demand.

The United States tariff law of 1930 provides a duty of \$4 per ton on crude barite and \$7.50 per ton on ground or otherwise manufactured.

#### PRODUCTION AND CONSUMPTION

Barite production in the United States started in Virginia about 1845. Production in Missouri began about 1860. European production probably began many years before it did in the United States. Many of the present uses for barite were unknown during the earlier years of production, hence the output necessary to meet the demand was small and variable. A gradual expansion of markets and a better knowledge of the chemical and physical properties of the mineral caused production to increase slowly. In the past 25 years consumption followed by production has advanced more rapidly. The United States, Germany and Great Britain are the principal producing countries. In some years German production exceeds that of the United States.

Fig. 1 shows graphically the gradual upward trend of production for the last 14 years if an average is considered, and the peaks and depressions balanced against each other. United States imports are also shown.

#### PROSPECTING, EXPLORATION AND MINING METHODS

Prospecting for barite is a rather simple and inexpensive operation. After the district where barite is known to occur has been explored in a



general way, the most favorable areas are selected for prospecting. Two methods are employed for residual deposits. In Missouri the choice is largely based on topography. A slender, pointed steel rod is driven into the ground, to bedrock if possible. If the rod penetrates barite the soft white mineral adheres to the rod when it is withdrawn. Chert and rock, if encountered, only scratch the rod without leaving a white coating. Test pits are sometimes sunk following the "rodding." In the Georgia (U. S.) residual deposits the practice is to sink test pits of circular section. The clay overburden stands very well without cribbing and the pits are often sunk to a depth of 30 ft. or more. They are only large enough for

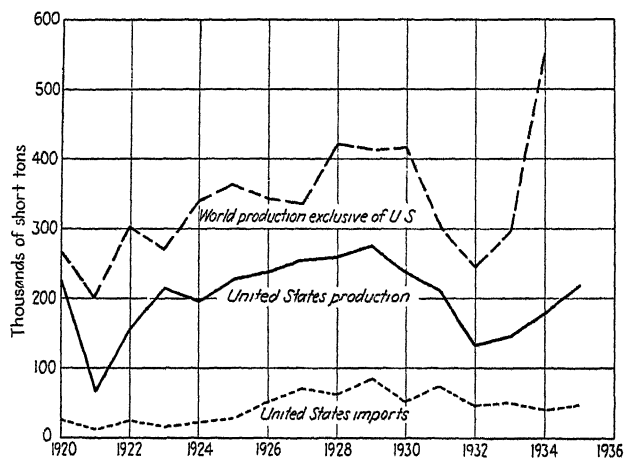


FIG. 1.—PRODUCTION OF CRUDE BARITE.

one man to work in with a short-handled pick and shovel. The excavated material is hoisted out by one man on the surface.

The prospecting of vein deposits is done in the usual way by trenching across the vein outcrop, sinking prospect shafts and tunneling when the topography permits. Elaborate and expensive methods such as diamond drilling are seldom resorted to.

*Mining Methods.*—Most of the United States production is from residual deposits, so the methods employed for these operations are of first importance. When labor is plentiful and low priced, hand mining is adopted. When it is scarce and higher priced, mechanical methods account for a greater amount. In the Missouri district both hand mining and mechanical or power-shovel mining are employed in working the residual deposits.

When barite is mined from veins or deposits in solid rock, the usual methods for such deposits are employed. The system employed depends on the topography, character of the wall rock, thickness and dip of the vein and the kind and amount of the associated minerals. Vein mining of minerals in hard rock has been fully described in other chapters.

*Hand Mining.*—The hand miner's or "tiff digger's" tools consist of a pick, shovel, sledge, small hammer with one end finished with a blade like a hatchet or chisel, hand windlass, rope, and ore bucket and an axe.

On the allotted area previously prospected, a pit or shaft is started where it is believed the best barite will be found. This shaft is  $4\frac{1}{2}$  to 5 ft. in diameter, usually circular, unless it has to be cribbed. The dirt is thrown out until the shaft becomes too deep. Many pits never go deeper. On the surface the dirt is worked over by pick and shovel and the lump barite picked out by hand and piled separately. The larger lumps are cleaned of clay with the aid of a hatchet or edge-faced hammer. When a good run of ore is struck, it is followed out horizontally as far as possible, until there is danger of the walls and roof caving in. Thus, in section, the shaft and excavation has the shape of an inverted mushroom. Occasionally the shafts reach a depth of 30 to 40 ft., but 10 to 15 ft. is more common. When the depth requires a windlass two or three men work together, one in the pit and one or two on the surface, hoisting and sorting. When one pit is worked out, another close by, often within 10 to 12 ft. of the other, is started and worked out in the same way.

Hand mining methods are best adapted to areas where the barite occurs in large masses or lumps and where the surface of the bedrock is uneven, making clean excavation with a power shovel difficult. The miner is invariably paid on a tonnage basis of barite recovered and cleaned.

In the central Missouri district, where the deposits are often on steeper hillsides, and deeper, the mining is done in open cuts with a level floor. The face of the bank is broken down by picks or barring from above. Sorting is done on the open-cut floor and the barite and clay are hauled out separately. Large boulders are left in place and mined around.

*Mechanical Methods.*—Mining with power shovels is sometimes done in Missouri where the deposit covers a fairly large area on gently sloping ground and where the surface of the bedrock is not too irregular or does not have many deep, narrow depressions. Areas previously worked over by hand are sometimes again excavated by a power shovel. Shovels are mounted on caterpillar treads and are of the full revolving type. Dippers are from  $\frac{3}{4}$  to  $1\frac{1}{2}$  cu. yd. in capacity. Gasoline or semi-Diesel engines are most common, but steam and electric are also used.

In excavating, the full depth of the deposit is taken at one cut and no overburden is previously stripped. The dirt is loaded into side-dump cars of about 5-yd. capacity and hauled to the washing plant by gasoline locomotives. On account of the uneven surface of the bedrock it is impossible for a power shovel to remove all the ore from the depressions. Where these are deep enough and frequent enough they are sometimes mined by hand after the shovel has worked over them.

In Georgia all mining is done with power shovels. The deposits are thicker than in Missouri and the slopes of the hill generally greater.

Sometimes the overburden is thick enough to remove and dispose of separately. If thin, it is excavated with the ore and sent to the washer. Adjacent deposits are sometimes connected by narrow stringers of pay dirt, permitting the shovel to cut through and follow from one to the other. Shovels are equipped with  $\frac{3}{4}$  to 1-yd. dippers. These load into small cars for haulage to the washer. Blasting of the face is sometimes resorted to where the bank is high, to prevent overhanging followed by possible caving and burying of the shovel.

#### PREPARATION FOR MARKET TESTS AND SPECIFICATIONS

Barite is sold or marketed in the form of crude lumps, jigged concentrates or table concentrates. Lump ore may be prepared from residual deposits or from vein deposits. It is essentially a product of hand sorting. With ore from veins the waste rock or gangue is sorted out with possibly some hand cobbing, either on platforms or picking belts. In one California mine where witherite occurs with the barite, the two minerals are sorted or separated by hand. Hand-mined ore from residual deposits, as in Missouri, is piled in the open to weather. The adhering clay dries, shrinks and falls loose from the barite or is scraped or chopped off with a hatchetlike tool. After drying the smaller lumps are shoveled into a rocker, a box about 20 in. wide by 32 in. long by 12 in. deep, fitted with a punched-plate or coarse wire screen bottom. The bottom is fitted with rockers, and as the box is rocked back and forth the clay falls through the screen. The screened barite and hand-cleaned lumps are then ready to haul to the nearest shipping point. Where the clay separates readily from the barite, the rocker is not used. After weathering, the operation of handling or loading with a fork into wagons or trucks, and again forking as loaded into railroad cars, removes the clay to the necessary limit.

Jigged and tabled barite concentrates are produced both from vein and residual deposits. Where there is no adhering or associated clay, standard practice for gravity concentration is practiced. With residual ores where most of the waste or gangue is clay, the jigging and tabling is always preceded by treatment in a log washer. Hand picking of the coarse, clean barite after passing through the log washer, is also practiced in some districts, notably Georgia.

A generalized flowsheet of modern practice in the Georgia field is shown in Fig. 2. This varies in detail to suit local conditions. For example, a jaw crusher may take the rake product of the log washer ahead of the coarse screen. However, there are usually very few compound or locked mineral grains or fragments in residual barite deposits, and these are usually broken up in the revolving coarse screen, as the barite is very friable. Barite washers usually have a capacity of 50 to 70 tons of "dirt" per hour. Of this often 75 per cent will be eliminated by the log washer in the first operation. Grizzly bars are usually placed 6 in. apart. Boulders

too large to pass are broken by hand or are thrown out if barren. Double washers 30 ft. long are almost standard size. The type and size of the jigs vary with conditions. The same remark applies to tables. There is a tendency towards the use of more tables in the Missouri district to prepare special grades of fine concentrates.

Most barite ores present no especially difficult dressing problems. Some residual ores, where the barite is coated with a layer of hard limo-

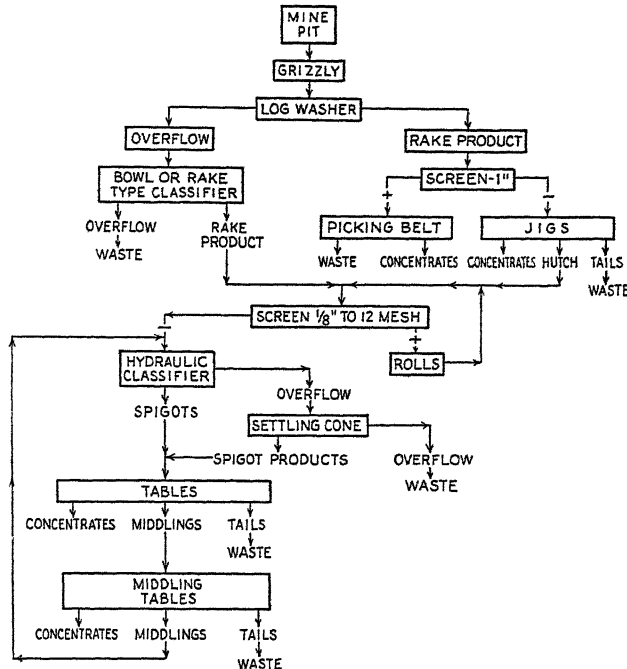


FIG. 2.—GENERALIZED FLOWSHEET, GEORGIA BARIUM CONCENTRATOR.

nite, present a problem because the removal of the coating by gravity methods is very difficult, as the gravity of the two minerals is close. Pyrite and sphalerite are difficult to separate by gravity methods. Galena is easily removed.

*Special Treatments.*—When the removal of limonite is essential, the limonite is sometimes removed by roasting and separation on a high-intensity magnetic separator. Another method is to leach the granular material, 14-mesh or finer, with dilute sulphuric acid. This does not completely bleach the barite but may lower the iron content sufficiently to meet special specifications for use in glass. In one operation in east Tennessee, where the limonite coating is excessive, the barite is crushed to a size larger than the granular product required and then calcined in a small rotary furnace. This causes the barite to decrepitate or break down without altering the size of the limonite grains. Then a fair separation

can be made on screens, which pass the finer barite and reject the limonite. In the Missouri district some of the barite is prepared for the glass trade by careful hand selection of hand-mined lumps low in iron. These are then crushed and screened to proper size.

Some barite occurring in veins and replacement deposits is intimately intergrown with quartz or other minerals, making gravity separation practically impossible. Some experimental work indicates that flotation may make a satisfactory concentrate from these ores.<sup>8</sup>

*Tests and Specifications.*—No standard tests or specifications for barite are in use. The most common specification used by a good part of the trade, however, is that the product shall contain 95 per cent  $\text{BaSO}_4$  and not over 1 per cent  $\text{Fe}_2\text{O}_3$ . A penalty is usually imposed if the ferric oxide exceeds 1 per cent and a premium allowed if it is lower. A premium is sometimes specified for a barium sulphate content in excess of 95 per cent. Purchase orders usually specify the size of the product and whether the barite is to be of the soft or hard variety. Barite for the glass trade is usually specified to contain not more than 0.1 per cent  $\text{Fe}_2\text{O}_3$ , not less than 96 per cent  $\text{BaSO}_4$  and to be crushed to pass a 16-mesh screen with not more than 5 per cent passing 100 mesh. This seems to be an arbitrary requirement, as some of the glass manufacturers are now asking for and taking a finely ground product.

#### MARKETING, USES AND PRICES

*Markets.*—The market outlet for crude barite or concentrates may be grouped under three general heads: (1) ground barite of various grades, (2) lithopone and (3) barium chemicals. The principal processing areas are in the Eastern States, Central States and California. All these areas consume barite for the three general uses mentioned. Some barite is also ground in South Carolina and Georgia. The producing districts of Georgia and east Tennessee market their barite largely on the Atlantic seaboard and in the Eastern States. Some of it moves north of the Ohio River. Imports are largely confined to the Atlantic seaboard. The Missouri barite is largely marketed in the North Central States, the St. Louis district and as far east as Pennsylvania. California production is mostly consumed or processed in California. This distribution of markets is largely controlled by transportation conditions.

Some of the largest consumers or processors of barite have their own mines, hence much of the production does not come into the competitive market. Most of the crude barite sold is marketed to consumers on the basis of contracts for a particular tonnage to be shipped over a definite period of time. A large producer may not be in the market for some time and then may place a contract for several thousand tons at one time, to supply his needs for several months. Minimum barium sulphate content and maximum iron oxide content are always specified. On some shipments the grain size is also specified.

*Uses.*—During the five-year period 1930–1934, of the total crude barite processed, amounting to  $1\frac{1}{4}$  million tons, both domestic production and imports, 19.1 per cent was ground, 58.2 per cent was used in lithopone and 22.7 per cent was used for the manufacture of barium chemicals.

Barite ground to various degrees of fineness is used as an inert filler in many products such as rubber, paper, oilcloth, plastics and resins. It is also used to weight textiles and leather. To some products it adds desirable properties. In others its function is mainly for weight and volume. All grades are used, from an off-color product to a white, acid-bleached one. Ground barite is also used in paint as a pigment and as an extender. A comparatively recent and important use is as a heavy medium in mud used in the drilling of deep oil wells where there are heavy gas pressures. A small amount of some suspending medium such as bentonite is often added to and ground with the barite. The use of barite for this purpose is covered by broad and comprehensive patents.

Granular barite, classed here under “ground,” is finding extensive use in the manufacture of glass. Small amounts added to the glass mix add desirable properties to some grades of glass, especially the molded forms. A low iron content is essential. Barite is used to some extent as a flux in brass melting, its function being mostly as a scavenger. It has also been proposed as a flux in the founding of gray iron.

Barite’s greatest single use is in the manufacture of lithopone. This is an intimate mixture of zinc sulphide and barium sulphate made by the coprecipitation of solutions of barium sulphide and zinc sulphate. It is mainly used as a white paint pigment but also finds considerable use as a filler. It contains approximately 70 per cent barium sulphate and 30 per cent zinc sulphide.

Barite is the raw material for the manufacture of most barium chemicals, although witherite is used when available. The initial step is the furnace reduction, with carbon to the soluble barium sulphide, known as “black ash,” or with calcium chloride to produce barium chloride. The principal barium chemical made is precipitated barium sulphate or *blanc fixe*. This is a whiter and finer grained product than it is possible to make mechanically from the natural barite. It is used as a filler, in paints and for special purposes. Precipitated barium carbonate is used in the ceramic industry and for making barium peroxide. Other important barium chemicals are the nitrate, chloride, hydrate, dioxide, peroxide, chromate and chlorate. Small amounts of metallic barium are used in some special alloys.

*Prices.*—Barite has always been and probably always will be a low-priced commodity, because of its wide distribution, ease of mining and concentrating, and because for many of its uses it is in direct competition with other low-priced minerals. Except for a few minor uses, it is not indispensable. Prices at point of production are governed largely by

cost of transportation to the marketing centers. The fluctuations in and the gradual upward trend of price as uses have expanded in the 30-year

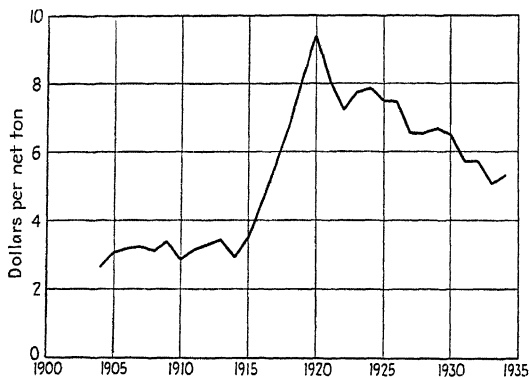


FIG. 3.—PRICE OF CRUDE BARITE, F.O.B. MINES.

period 1904–1934 are shown graphically in Fig. 3, plotted from statistics by the U. S. Geological Survey and U. S. Bureau of Mines.<sup>7</sup>

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## CHAPTER V

### BAUXITE\*

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**B**AUXITE is known mainly as the ore from which aluminum is smelted, but it has large use also in the manufacture of artificial abrasives and as a basis for certain chemical industries. A small amount is used for refractories and for other purposes.

#### COMPOSITION AND PROPERTIES

*Composition.*—Dana and others give the mineral formula of bauxite as  $\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ , and the composition  $\text{Al}_2\text{O}_3$ , 73.9 per cent;  $\text{H}_2\text{O}$ , 26.1 per cent. It has been definitely shown, however, that bauxite does not exist as a specific mineral but rather as a rock. The term is now used synonymously with aluminum ore and it embraces gibbsite (hydrargillite or alpha trihydrate),  $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$  ( $\text{Al}_2\text{O}_3$ , 65.4 per cent;  $\text{H}_2\text{O}$ , 34.6 per cent); boehmite (alpha monohydrate),  $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$  ( $\text{Al}_2\text{O}_3$ , 85 per cent;  $\text{H}_2\text{O}$ , 15 per cent), diaspore (beta monohydrate),  $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$  ( $\text{Al}_2\text{O}_3$ , 85 per cent;  $\text{H}_2\text{O}$ , 15 per cent) and mixtures in various proportions of any two of them. Bauxite of the Mesozoic and Tertiary in Europe is predominantly a mixture of gibbsite and boehmite, subordinately a mixture of boehmite and diaspore, or gibbsite and diaspore. The bauxite of North and South America, tropical Africa and Asia, consists largely of gibbsite. Corundum ( $\text{Al}_2\text{O}_3$ ) is not included under bauxite, although gradations from bauxite to emery or corundum exist.

All bauxite, irrespective of the aluminum minerals composing it, contains certain impurities, including silica, in the form of clay minerals (kaolinite, halloysite, etc.) or quartz, iron oxide (as hematite or goethite), titania (as leucoxene or rutile), iron sulphide (as pyrite or marcasite), iron carbonate (siderite), and calcium carbonate (as calcite); the last three mentioned in minor amount and of local occurrence only. Bauxite belongs to a group of partly consolidated materials called laterites, formed by surface weathering. This group includes also impure siliceous and ferruginous bauxite and siliceous and aluminous iron ores and manganese ores.

\* Through the courtesy of the McGraw-Hill Book Co. much of this article was abstracted from the chapter on Ores of Aluminum, by E. C. Harder, in *The Aluminum Industry*, by J. D. Edwards, F. C. Frary and Z. Jeffries, New York, 1930. Other references are given in the bibliography at the end of this chapter.

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*Properties.*—Gibbsite is white or light shades of gray, cream or pink. Its hardness is 2.5 to 3.5; specific gravity, 2.3 to 2.4; crystal system, monoclinic, crystals tabular parallel to 001, often fibrous, concretionary, stalactitic; luster, vitreous to pearly; generally translucent; cleavage, parallel to 001; disassociation temperature, approximately 300° C.

Boehmite, a new mineral described by J. Boehm in 1925, confirmed and named by J. de Lapparent in 1930, has some properties not yet definitely determined. It occurs in grayish, brownish and reddish shades. Its hardness is between that of gibbsite and diaspore; specific gravity, similar to diaspore; crystal system, orthorhombic, best developed along 110 and 001; angle between the faces *m* near 63°; cleavage, parallel to 010; X-ray pattern distinctly different from that of diaspore.

Diaspore is white or light shades of gray or brown. Its hardness is 6.5 to 7; specific gravity, 3.3 to 3.5; crystal system, orthorhombic, crystals prismatic, flattened, parallel to 010; luster, pearly to vitreous; transparent to translucent; birefringence high; cleavage, parallel to 010; disassociation temperature approximately 500° C.

Bauxite as it occurs in nature rarely exhibits the distinct characteristics of its constituent minerals. These become apparent only by examination of thin section under the microscope, by the determination of heating curves, by chemical analysis or by X-ray tests. Not all the constituents in bauxite are recognizable even by such methods and it is therefore believed that much of the alumina is present in the form of colloidal hydrogels, with a variable combined water content. Bauxite is a medium soft to hard rock, has a fine-grained cellular, porous or compact structure, a conchoidal or uneven fracture and ranges in color from light gray, cream, pink or yellow to dark brown and dark red. In many localities the bauxite is characterized by an oolitic or pisolitic texture, the oolites ranging in size from less than  $\frac{1}{20}$  in. to  $\frac{1}{2}$  in. or more. The oolites may have a concentric texture or they may be homogeneous. Small oolites may occur grouped together within larger ones. The color of the oolites generally differs somewhat from that of the matrix—usually, but not always, being darker. There is much bauxite that does not show oolitic texture but has an even-grained, homogeneous or claylike appearance. Some is porous or cellular. In many places oolitic and nonoolitic bauxite occur together in the same deposit.

#### MODE OF OCCURRENCE

Bauxite deposits occur in a number of different geologic associations but most of them can be grouped into four main classes:

1. Blanket deposits occurring at or near the surface in horizontal, tilted, or undulating sheets and lenses.

2. Beds and lenslike deposits occurring at definite stratigraphic horizons interlayered with sediments or between sediments and igneous rocks.

3. Pocket deposits or irregular masses enclosed within limestone or clay.

4. Detrital deposits formed by the mechanical breaking up, transportation and redeposition of the material from deposits of the other three types.

These four types of deposits are closely related to each other and are in many cases gradational. There is a strong possibility that the first three have a somewhat similar origin.

The table on page 114 shows the general age relations of the principal bauxite deposits of the world.

*Blanket Deposits.*—Horizontal blanket deposits of bauxite or laterite are commonly found in tropical or semitropical regions, where they occur on base-leveled surfaces. Some of them are at low elevations and others occur as cappings on extensive plateaus or on flat-topped hills representing outliers or remnants of such plateaus. The elevated bauxite-bearing plateaus are former base-leveled surfaces that have been raised and subjected to erosion subsequent to the original peneplanation and formation of bauxite. In many places the laterite or bauxite deposits are at the surface, covered only by sparse vegetation; elsewhere, varying thicknesses of soil cover them. Generally the surface and upper portion of the deposits is hard and indurated owing to infiltration and cementation by iron oxide or siliceous material. With depth, it becomes softer and at the bottom it passes rather abruptly into the underlying residual clay. Locally small, irregular pipes or networks of pipes of bauxite project downward into the residual clay from the lower surface of the bauxite layer.

Blanket deposits of bauxite or laterite range in thickness from less than 1 ft. to 75 ft. or more; the average is from perhaps 5 to 25 ft. Horizontally blanket deposits may extend for a few hundred feet or for several miles. The entire layer, except for variations at the immediate surface or at the base, may be fairly uniform and may consist of high-grade bauxite or of siliceous or ferruginous bauxite. On the other hand, however, there may be variations both vertically and horizontally, with the different phases lying in well marked layers or grading into each other laterally or being irregularly intermixed.

*Interlayered Deposits.*—Most layers and lenses of bauxite or laterite interbedded with sediments or lying between sediments and igneous rocks are what might be termed "fossil blanket deposits." They represent surface deposits of past geologic ages that have been submerged subsequent to their formation and have had deposited upon them successive beds of later sediments. As far as texture and composition are concerned, interbedded bauxite deposits in general resemble surface deposits, except that they have a tendency to be harder, more compact and more indurated, owing, no doubt, to the weight of the overlying sediments. They show

*General Age Relations of Principal Bauxite Deposits*

Pleistocene and Recent	Tropical lateritic bauxite of the Guianas, Brazil, French West Africa, Gold Coast, Nyasaland, India, Dutch East Indies, Australia
Cenozoic	
Pliocene	
Miocene	German bauxite (Vogelsberg Mountains)
Oligocene	Irish bauxite (County Antrim)
Eocene	
Upper	
Middle	
Lower	United States bauxite—underlain by lower Eocene or Lower Cretaceous
	Dalmatian bauxite—underlain by lower Eocene and Upper Cretaceous
	Montenegrin bauxite (Bar and Ulcinj)—underlain by Upper Cretaceous
	Istrian bauxite—underlain by Upper Cretaceous
	Spanish bauxite—underlain by Eocene and Rhaetic
	Kashmir deposits (Jammu)—underlain by Jurassic
	Hungarian bauxite—underlain by Rhaetic
Mesozoic	
Cretaceous	
Upper,	
Danian	
Senonian	Greek bauxite—underlain by Jurassic
	French bauxite (Herault)—underlain by Jurassic
Middle,	
Turonian	
Cenomanian	
Albian	Italian bauxite (Central Apennines)
	Montenegrin bauxite (Kotor and Niksic)—underlain by Lower Cretaceous
	French bauxite (Var)—underlain by Urgonian, Neocomian and locally Jurassic
Lower,	
Aptian	
Urgonian	French bauxite (Ariege)—underlain by Jurassic
Neocomian	Rumanian bauxite (Bihar)—underlain by Malm
Jurassic	
Malm	
Dogger	
Lias	
Triassic	
Rhaetic	Croatian bauxite—underlain by Middle Triassic
Keuper	
Middle	
Lower	Chinese diaspore (Poshan)—underlain by "Permo-Carboniferous"
Paleozoic	
Upper Carboniferous	Missouri diaspore—underlain by upper Carboniferous
Lower Carboniferous	Russian bauxite (Tikhvin)—underlain by Devonian
Devonian	Russian bauxite (Ural)—in Middle and Lower Devonian

the same variations in grade, however, and have the same impurities. The overlying rocks may be limestone, shale or sandstone; the underlying rocks may be igneous or sedimentary. In many places rocks with which the bauxite layers and lenses are interstratified have suffered deformation with resultant folding and faulting of the bauxite layers. In places where the deformation has been pronounced, as in parts of France, Rumania and Greece, there has been noted the partial alteration of gibbsite or boehmite to diaspore. Where the deformation has been extreme, as in some of the Aegean Islands, corundum (emery) has developed.

*Pocket Deposits.*—Pocket deposits occur in many places associated with interbedded bauxite layers and lenses. They represent irregularities in the surface upon which the bauxite was formed. In many places bauxite pockets are directly connected with bauxite layers or lenses as extensions from their lower surfaces into pre-existing depressions on the upper surface of the stratigraphically underlying rock, commonly limestone, but in places clay. Elsewhere, pockets are scattered irregularly or occur in groups unconnected with bauxite layers or lenses which, if they ever existed, have been removed by erosion.

The pocket deposits of bauxite generally show sharp contacts with the enclosing limestone or clay, although some deposits in clay show gradational margins of bauxitic clay high in alumina. Similarly, bauxite pockets in limestone may be directly in contact with the limestone or may be separated from it by masses of residual earth, such as terra rossa, derived from limestone decay. The shape of the pockets is irregular; some are approximately equidimensional but most are irregularly pod-shaped. The manner in which they cut across the bedding of the enclosing rocks, and their arrangement in groups, suggests deposition of the protore as fillings of solution caverns.

*Detrital Deposits.*—Detrital bauxite deposits originate from other pre-existing bauxite deposits, and may be talus accumulations, stream gravels or sands, or more or less consolidated low-level surface layers.

#### ORIGIN

The hypotheses that have been advanced to explain the origin of bauxite may be grouped under three main headings: (1) chemical sedimentation, (2) weathering *in situ*, (3) detrital deposition.

Many geologists have considered it necessary to explain the formation of bauxite deposits by processes of solution and redeposition. Clays or other aluminum-bearing rocks were looked upon as the source of the aluminum. As solvents, various acids and alkalis were taken into consideration—for instance, nitric acid, derived from bacterial activity, or from lightning discharges; sulphuric acid resulting from the decomposition of sulphides and sodium hydroxide or sodium carbonate, obtained by decomposition of various rocks.

In recent years the role of weathering processes in the formation of bauxite and laterite has been clearly recognized, and the general opinion today is that most bauxite deposits, rather than being the result of solution and redeposition of aluminum, are residual products resulting from the intensive weathering of aluminum-bearing rocks, involving the solution and removal of constituents other than aluminum. This process is called lateritization, and the original rocks most commonly involved are syenite, granite, basalt, diabase, crystalline schists, limestone, shale and clay. It is evident, therefore, that the bauxite itself may be somewhat younger geologically than the bedrock with which it is associated.

Certain bauxite deposits of minor importance are believed to be of detrital origin, being the result of mechanical concentration of bauxite fragments from previously existing bauxitic materials and in places their partial consolidation by infiltration of cementing substances.

#### DISTRIBUTION OF DEPOSITS

Although bauxite deposits occur abundantly in many parts of the world, the mining of bauxite in important quantities is confined to relatively few countries. Roughly, in the order of their importance, the principal bauxite-producing countries, not including Russia, are France, United States, Dutch Guiana, British Guiana, Hungary, Italy and Yugoslavia. Russia has entered the ranks of important producers only within the last three or four years. Minor quantities of bauxite are produced in Greece, northern Ireland, India, Rumania, Spain, Australia and Germany. France produces roughly one-third of the world's bauxite, the United States approximately one-fifth, Dutch Guiana, Hungary and British Guiana, each about one-tenth, Italy about one-fifteenth, and Yugoslavia about one-twentieth (Fig. 1).

*France.*—The principal bauxite deposits of France occur in the departments of Var and Herault; minor deposits in the departments of Bouches de Rhone and Ariege. About four-fifths of the bauxite produced in France has come from the Var district, and three-quarters of the remainder from Herault. Besides furnishing the total requirements of France, these deposits have supplied a considerable portion of the bauxite used elsewhere in Europe, particularly in the United Kingdom and Germany. Bauxite was discovered in France in 1821, near Les Baux in Bouches du Rhone, from which locality it receives its name. The first recorded production occurred there in 1873.

The French bauxite is mostly of the dark red, high-iron type. This variety ordinarily contains 56 to 59 per cent  $\text{Al}_2\text{O}_3$ , 20 to 25 per cent  $\text{Fe}_2\text{O}_3$  and 2 to 5 per cent  $\text{SiO}_2$ . Small amounts of white and gray bauxite are produced, the former containing 60 to 70 per cent  $\text{Al}_2\text{O}_3$ , about 4 to 12 per cent  $\text{Fe}_2\text{O}_3$  and 8 to 16 per cent  $\text{SiO}_2$ , and the latter having a variable composition ranging between the red and the white bauxite. All

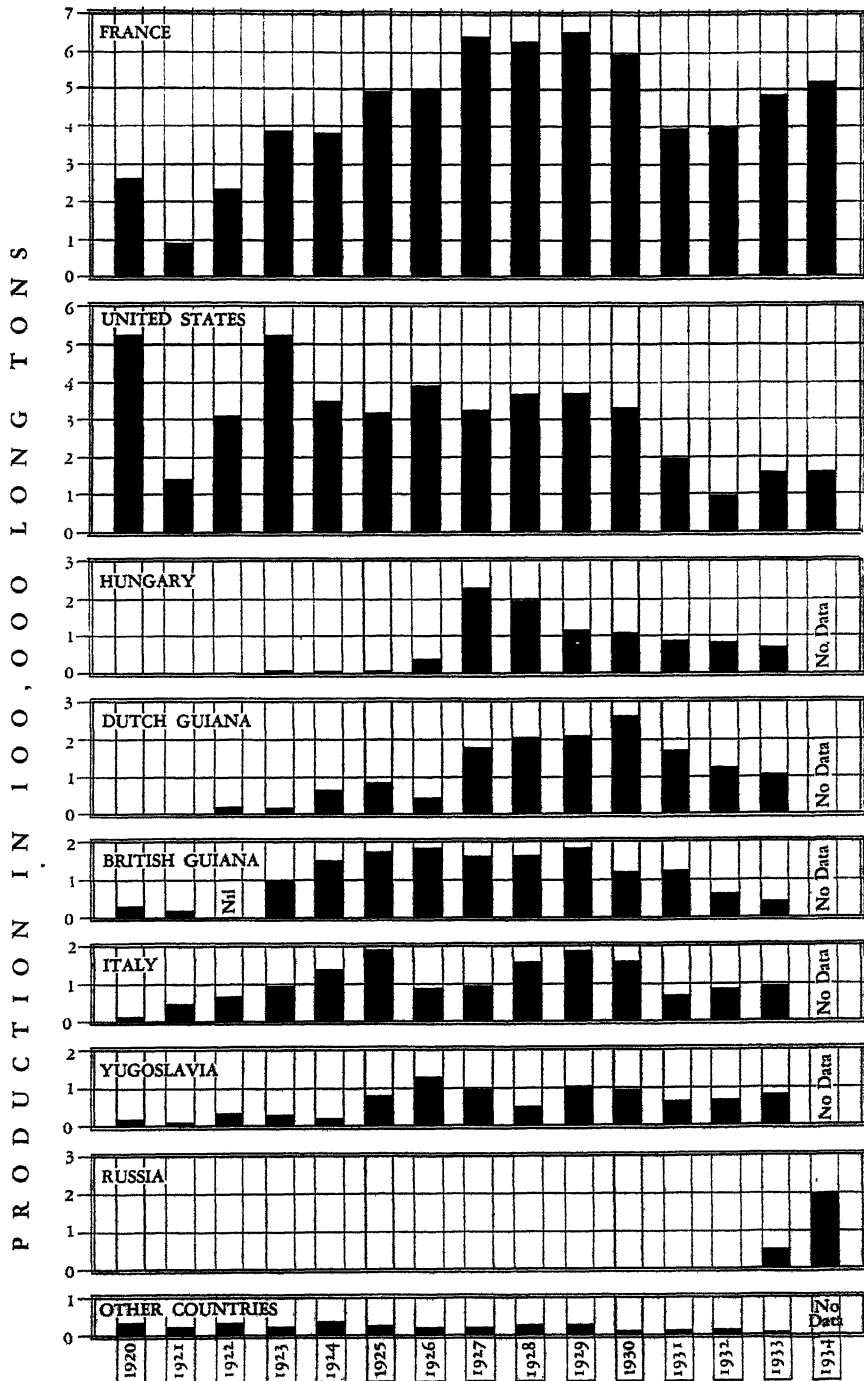


FIG. 1.—BAUXITE PRODUCTION BY COUNTRIES, 1920 TO 1934.

three types contain 12 to 14 per cent combined water. The different varieties all occur in association with limestone as pockets or interlayered beds and lenses. They vary in relative abundance in the different districts.

*United States.*—Bauxite was first discovered in the United States near Rome, Ga., in 1883. The first production occurred in Georgia in 1889 and in Arkansas 10 years later. Deposits occur in Arkansas, Georgia, Alabama, Tennessee and Mississippi. The Arkansas deposits have furnished about 90 per cent of all the bauxite produced in the United States. They are alterations of nepheline syenite and clay and are overlain by Eocene clays. The deposits in northern Georgia, northern Alabama and Tennessee occur as pockets in clays; those in southern Georgia and southern Alabama are in the form of beds and lenses interlayered in Eocene sediments.

The bauxites of the United States differ from those occurring in Europe in that usually they contain a relatively small amount of iron oxide and carry a high content of combined water. On an average they contain 56 to 59 per cent  $\text{Al}_2\text{O}_3$ , from less than 2 to about 6 per cent  $\text{Fe}_2\text{O}_3$ , from 5 to 8 per cent  $\text{SiO}_2$  and approximately 29 to 31 per cent combined water. In general the deposits in Georgia, Alabama and Tennessee are lower in iron oxide and higher in silica than those in Arkansas. The bauxite deposits of Mississippi are of low grade and under present conditions are considered uncommercial.

There occur also in the United States, in central Missouri, numerous but small and scattered deposits of diaspor, which have furnished an important production in recent years, being used mainly in the manufacture of aluminous refractories. The Missouri diaspor, which forms small irregular masses in flint-clay pockets, varies in alumina content from 50 to more than 70 per cent, depending on the amount of intermixed clayey materials. The low-grade diaspor is known as burley clay.

*Dutch Guiana.*—Bauxite was discovered in Dutch Guiana about 1915 and the first shipments were made in 1922. Most of the ore is exported to the United States for manufacture of aluminum. It is found along Surinam, Cottica and Coermotibo Rivers, south and southeast of Paramaribo, and about 15 to 25 miles inland from the coast. It is of the tropical lateritic variety and occurs mostly as surface deposits, forming low hills and ridges in the coastal lowlands, with only a few feet of soil overburden. The Dutch Guiana bauxite is of high grade, containing 57 to 59 per cent  $\text{Al}_2\text{O}_3$ , 5 to 7 per cent  $\text{Fe}_2\text{O}_3$ , 1.5 per cent to 2.5 per cent  $\text{SiO}_2$  and 31 to 32 per cent combined water.

*Hungary.*—The Hungarian bauxite deposits occur in an area north of Lake Balaton and extend thence northeastward toward Budapest. Most of the ore produced thus far has come from the Vertes Mountains, in the northeastern part of the belt. The bauxite ranges from yellow to brown and red in color, and that of commercial grade contains 57 to 60 per

cent  $\text{Al}_2\text{O}_3$ , 12 to 20 per cent  $\text{Fe}_2\text{O}_3$ , 2 to 7 per cent  $\text{SiO}_2$  and 14 to 16 per cent combined water. However, a large proportion of the material in the deposits is of low grade and must be removed along with the commercial ore, increasing the mining expense. The Hungarian bauxite is similar to that of the Mediterranean region in being associated with limestone in pockets and lenses. It was first exploited in 1925 and since that time has furnished a large proportion of the bauxite used in Europe for aluminum manufacture.

*British Guiana.*—Bauxite was first exploited in British Guiana in 1914 and shipments began in 1917. The principal deposits are in the Demerara and Berbice River regions, from 65 to 125 miles south and southeast of Georgetown. They occur as horizontal or slightly tilted beds and lenses, in places outcropping along the rivers and small creeks and elsewhere overlain by a varying thickness of recent coastal deposits. In the exploited areas this overburden of sand and clay ranges up to 100 ft. or more in thickness and overlies 15 to 25 ft. of bauxite. British Guiana bauxite contains 59 to 61 per cent  $\text{Al}_2\text{O}_3$ , 1 to 2.5 per cent  $\text{Fe}_2\text{O}_3$ , 2.5 to 4 per cent  $\text{SiO}_2$  and 30 to 32 per cent combined water. It is used mainly for manufacture of aluminum but because of its low content of iron oxide it is also in great demand for the manufacture of chemicals and refractories. Most of it is exported to Canada and the United States but considerable tonnages are shipped also to the United Kingdom and continental Europe.

*Italy.*—Important deposits of bauxite occur in the Istrian Peninsula south and southeast of Trieste. Most of them are in the form of pockets in limestone, abundant and widely scattered. The mining of bauxite in Istria on an important scale began during the World War and these deposits have since furnished not only the Italian requirements but important tonnages annually for export to other European bauxite-consuming countries. Large but relatively low-grade bauxite deposits occur in the central Apennine region of Italy and periodically have furnished a small production.

The Istrian bauxite is of two main types; one, dark red, similar to that produced in southern France and the other a yellowish variegated ore. The former generally contains between 55 and 58 per cent  $\text{Al}_2\text{O}_3$ , 22 to 25 per cent  $\text{Fe}_2\text{O}_3$ , 2 to 4 per cent  $\text{SiO}_2$  and 12 to 14 per cent combined water. The latter commonly carries 60 to 65 per cent  $\text{Al}_2\text{O}_3$ , 10 to 15 per cent  $\text{Fe}_2\text{O}_3$ , 5 to 9 per cent  $\text{SiO}_2$  and 12 to 14 per cent combined water. The different varieties may occur together in the same deposit or they may form separate deposits.

*Yugoslavia.*—The Yugoslavian bauxite deposits occur mainly in the provinces of Dalmatia, Herzegovina and Montenegro. Only those of Dalmatia have been extensively exploited, as the Herzegovinian deposits have been opened up only within the last year, while the Montenegrin deposits are mostly of low grade. Most of the bauxite mined in Dalmatia has come from Drnis district, about 50 miles north-



east of Sibenik, but there are also mines in the Obrovac district and on some of the coastal islands. The Yugoslavian bauxite is of the dark red variety common elsewhere in southern Europe but differing from other European bauxite in that it contains a higher percentage of combined water. It contains approximately 50 to 54 per cent  $\text{Al}_2\text{O}_3$ , 20 to 24 per cent  $\text{Fe}_2\text{O}_3$ , 1 to 3 per cent  $\text{SiO}_2$  and 18 to 22 per cent combined water. Bauxite mining began in Yugoslavia during the World War, and much of the bauxite used by the Central Powers during this period was obtained from this source.

*Russia.*—Since the beginning of the first Five-Year Plan, Russia has come to the front as a producer of bauxite. The bauxite mined thus far has been derived from the Tikhvinsk district southeast of Leningrad, but during the last few years there have been reports of important bauxite discoveries in the Ural Mountains north and south of Sverdlovsk. The Tikhvinsk bauxite contains about 35 to 54 per cent  $\text{Al}_2\text{O}_3$ , 15 to 20 per cent  $\text{Fe}_2\text{O}_3$ , 10 to 20 per cent  $\text{SiO}_2$  and 10 to 20 per cent combined water. The ore produced at present is being used mainly in the manufacture of aluminum at the new Russian smelting plants at Volkhov and on the Dnieper River. It is of very low grade, however, and would be considered uncommercial except from the nationalistic viewpoint.

*Greece.*—Although not as yet a large producer of bauxite, Greece contains important deposits, some of which are in process of development at the present time. The principal Greek bauxite occurs in the region north of the Gulf of Corinth, but deposits are also found in Euboea and on certain islands in the Aegean. On account of their proximity to the coast, they are well situated for exploitation. The Greek bauxite, although of the dark red ferruginous type, common in Europe, is harder and more refractory. It contains about 56 to 59 per cent  $\text{Al}_2\text{O}_3$ , 16 to 20 per cent  $\text{Fe}_2\text{O}_3$ , 3 to 7 per cent  $\text{SiO}_2$  and 13 to 15 per cent combined water.

*Netherlands Indies.*—The bauxite deposits of the island of Bintan, near Singapore, have been developed within the last three years to supply the new Japanese aluminum industry. Some ore has recently been shipped to Germany, analyzing approximately 55 per cent  $\text{Al}_2\text{O}_3$ , 2.50 per cent  $\text{SiO}_2$ , 15 per cent  $\text{Fe}_2\text{O}_3$  and 29 per cent combined water.

*Other Countries.*—The bauxite deposits of Rumania are similar to those of Greece. Because of their remoteness from markets, they are not being exploited. The bauxite of British India is of two types, that occurring in the Central Provinces and in southern and southeastern India, which is of the ordinary, lateritic type, and that of the Jammu district, Kashmir, in northwestern India, which is of the diaspore type. Indian bauxite is being shipped in small amounts to Japan and Europe. The bauxite of northern Ireland is a low-grade clayey type, locally high in iron oxide. Small and relatively unimportant bauxite deposits have been exploited in Spain, Germany and Australia.

Large and numerous deposits of bauxite occur in various tropical and subtropical countries but have remained undeveloped mainly because of their remoteness from bauxite-consuming centers. Such are the deposits of French Guinea, Gold Coast and elsewhere in West Africa, those of Nyasaland in East Africa, and of Minas Geraes, Brazil.

#### POLITICAL AND COMMERCIAL CONTROL

Like most mineral raw materials, bauxite supplies are unequally distributed among the important world powers, particularly from the viewpoint of the aluminum industry in these powers.

The United States, as the leading producer in the world of metallic aluminum, aluminum chemicals and aluminous abrasives, is insufficiently supplied with bauxite reserves. Although the United States deposits furnish an important production, a large tonnage of bauxite is annually imported and little is exported, except in the form of refined products. In spite of this situation, there is an import duty of \$1 per ton on bauxite brought into the United States.

Germany, as the next largest producer of metallic aluminum and aluminum products, has practically no commercial bauxite within its borders. The German bauxite requirements are obtained mainly from Hungary, and subordinately from France, Italy and Yugoslavia. About a year ago a substantial import movement began from the Netherlands Indies. France and Italy, during the last year or two, have placed restrictions on the exportation of bauxite, making it increasingly difficult for Germany to obtain supplies from these sources.

France is well supplied with bauxite reserves and may rely on its own deposits to furnish the requirements of the domestic aluminum and other bauxite-consuming industries for many years to come. France imports no bauxite from abroad, but has exported substantial quantities to United Kingdom, Germany and elsewhere.

Although there are no bauxite deposits in the United Kingdom itself, the British Empire is well supplied with bauxite, both developed and undeveloped. Of these, among the most important are those in British Guiana, which supply the Canadian aluminum industry, and are furnishing increasing quantities to the United Kingdom. Other important, but as yet largely undeveloped, deposits occur in British India, Gold Coast Nyassaland. Most of the bauxite requirements of the United Kingdom have in the past been imported from southern Europe, chiefly from France. It may be expected, however, that with the present tendency towards economic nationalism these sources will become more restricted and the United Kingdom will have to depend increasingly upon the bauxite resources of the British Empire.

Norway and Sweden, with their important aluminum industries, are dependent entirely upon imported bauxite. In the past bauxite supplies

have been obtained mainly from France and Italy, but it is conceivable that in the not far distant future Scandinavian aluminum producers will have to go farther afield for their bauxite requirements.

Switzerland, an important producer of aluminum, has no bauxite deposits. Raw materials for its plants have been obtained largely from France in the form of alumina manufactured from French bauxite.

Italy has substantial bauxite reserves, which will be able to supply the domestic aluminum and other bauxite-consuming industries for some time in the future.

Hungary produces aluminous cement and also a small amount of aluminum in a recently built plant. A relatively minor part of its bauxite output, however, is used internally.

Russia, with its rapidly expanding aluminum industry, has a dearth of aluminous raw materials. The presently developed bauxite deposits, although uneconomical, will serve to supply the domestic consuming industries for a brief period, but it is clear that the time is approaching when Russia will find it necessary to go across its borders for bauxite supplies if its alumina and aluminum plants are to be kept operating.

Japan has ambitions to become independent with regard to its aluminum requirements. However, Japan has no bauxite deposits and it is not likely that large deposits will be discovered in territories under its control. Until recently the small aluminum-producing plants in Japan were using Korean alunite and Manchurian clays. Small amounts of European and British Indian bauxite have been imported and bauxite deposits in the Netherlands Indies have been developed, looking to a future Japanese market. An important movement from this latter source to the new Formosa smelter has already begun.

A number of countries possess important bauxite deposits but have no aluminum or other important bauxite-consuming industries. Thus the Netherlands has large bauxite deposits in Dutch Guiana and in the Netherlands Indies, the former furnishing an important part of the United States requirements and the latter, as already mentioned, being developed for the expanding Japanese industry. Yugoslavia has exported important tonnages of bauxite annually to Germany and elsewhere, but has consumed very little within its own borders. Brazil also has large bauxite deposits but has negligible bauxite-consuming industries.

#### PRODUCTION AND CONSUMPTION

Although bauxite was discovered in France in 1821 there is no record of any production until 1873, when 200 tons was mined. At the end of the century the output had not reached 50,000 tons. Thereafter, however, bauxite mining progressed rapidly and in 1913 more than 300,000 tons was produced. In the United States the first bauxite production dates from 1889, when 728 tons was mined in the Rome district. The Arkansas bauxite mines began producing in 1899, with an output of 5045 tons in

that year. Until the World War started, more than 95 per cent of the bauxite produced annually came from the mines of France and the United States. During the war, however, the necessity for the opening up of new bauxite deposits became apparent and while the deposits of Istria and Dalmatia were being developed by the Central Powers, the Allies turned their attention to the newly discovered deposits of British and Dutch Guiana. British Guiana mines started shipments in 1917 and the Dutch Guiana mines in 1922. Somewhat later bauxite was discovered in Hungary and was developed for the benefit of the German aluminum plants. A very recent entrant into the ranks of producers is Russia, with a spectacular rise in production in 1933 and 1934.

#### PROSPECTING, EXPLORATION AND MINING

*Prospecting.*—Although bauxite deposits were formed under conditions existing in warm climates, they are not necessarily confined to tropical or subtropical regions, owing to variations in climatic conditions in past geologic times. Thus the Arkansas bauxite deposits occur at latitude 35° N., those of southern Europe between 40° and 50° N. and those of the Tikhvinsk region in Russia at latitude 60° N. Bauxite deposits are usually discovered from outcrops and fragments of float. Rock associations, however, may serve as a guide, bauxite being commonly found with clay, limestone or certain igneous rocks, such as syenites, feldspathic schists and basalts. Probably the most striking association of bauxite is with old land surfaces. Thus bauxite deposits in the older rocks occur along unconformities representing long time intervals, while those of recent origin occur on moderately level, peneplained surfaces that have been undisturbed for a long period of time.

*Exploration.*—Once found by outcrops, bauxite deposits are explored by drilling. It is generally preferable to use hand drills, as most bauxite deposits occur in remote regions where transportation is difficult. Moreover, many bauxite deposits are found at relatively shallow depths and power drills are unnecessary. Core drills usually are not suitable, as bauxite and the associated sediments are relatively soft and do not yield satisfactory cores. It is best to perform the exploration by drills in two stages, the early drilling, widely spaced, and merely for the purpose of determining the quality and estimating approximate tonnages, and the later drilling at close intervals to serve as a guide to mining. A few test pits should be sunk on all deposits to supplement the drilling, mainly with the object of securing samples for washing and other beneficiating tests.

*Mining Methods.*—The methods used in mining bauxite differ somewhat in different countries and with different types of deposits. In deposits occurring in the form of extensive surface blankets or in beds and lenses interlayered with unconsolidated sediments, such as are being mined in Arkansas and in northern South America, quarrying is a common method of mining. The overburden, where present, is removed by

hand or by scrapers or, where thick, by power shovels or draglines, and the upper surface of the bauxite layer is thoroughly cleaned by means of picks and shovels. Holes for blasting are then drilled into the bauxite bed vertically, starting at the surface and penetrating into the lower portion of the bauxite layer. Generally such holes are drilled in series parallel to the existing quarry faces. After blasting, the broken bauxite is picked up by power shovels, loaded on mine cars and transported by rail to beneficiation plants.

Where the overburden is too deep to be removed by stripping, or where lenses and layers of bauxite are interlayered with solid rock, such as limestone in the European fields, and the attitude of the enclosing rock is such that the deposits reach considerable depths, quarrying methods are used only along the outcrops and underground mining is resorted to at depths. The underground mining is advanced by a series of drifts and crosscuts and caving is employed in retreating. The bauxite is removed from the workings in mine cars, through tunnels, shafts or inclined planes, according to the form of the deposit and the method of working, and is transported from the mines to railway stations or seaports by motor lorries. European bauxite is not subjected to beneficiation.

Pocket deposits of bauxite, such as occur enclosed in limestone at various places in southern Europe and in clay in southeastern United States, are mined by open quarrying methods. Bauxite pockets are usually of limited extent and the mining is mainly by hand with the use of picks, shovels and wheelbarrows. In large pockets mine cars may be used. The bauxite is raised from the open pits along inclines or by elevators, and transported to railway stations by motor lorries. At several localities in Europe where the conditions permit, aerial cableways are in use between mines and loading stations.

#### PREPARATION FOR MARKET, TESTS AND SPECIFICATIONS

*Preparation.*—In Arkansas, British Guiana and Dutch Guiana the bauxite is beneficiated before marketing. In Europe the bauxite is shipped direct to consuming plants in the crude state as mined. In the United States and in the Guianas the crude bauxite, as mined, consists of fines and fragments ranging in size up to a foot or more and contains 12 to 15 per cent free moisture. This crude bauxite is crushed to a maximum size of  $2\frac{1}{2}$  to 3 in. and is then washed to remove the clay and other impurities present. The washing is generally performed in trommel-rake washers but locally vibrating screens are used to supplement these.

As the washed bauxite contains about 15 to 18 per cent moisture, it is dried before shipment, in rotary kilns that remove all except 1 to 3 per cent of the free moisture. The rotary kilns range from 60 to 120 ft. in length and 6 to 8 ft. in diameter, depending upon the size of the operation.

They are lined with firebrick. The temperature of the bauxite leaving these kilns is about 200° F. and 35 to 40 tons per kiln hour may be produced. This is the ordinary crushed, washed and dried bauxite consisting of mixed lumps and fines that is marketed for the manufacture of aluminum and for chemical, abrasive and refractory purposes. The main reason for drying the bauxite at the mines is to save transportation costs, but drying is also important for the fabricating plants where the bauxite is to be consumed, as the fine grinding necessary for further treatment during processing requires a dry material.

It is desirable, when bauxite is used for abrasive and refractory manufacture, that the combined water as well as the free moisture should be removed. Therefore some bauxite producers are equipped with rotary kilns designed especially for calcining. Such kilns range up to 150 ft. or more in length and have heavy refractory linings. The calcining process yields only about 3 to 4 tons of bauxite an hour. The calcined mineral leaves the kiln at a temperature of 1700° to 1800° F.

*Tests and Specifications.*—The bauxite to be used in the various consuming industries differs considerably in composition. To be suitable for the manufacture of metallic aluminum and aluminous abrasives, bauxite should be as low as possible in silica. Preferably, it should contain less than 2.5 or 3 per cent  $\text{SiO}_2$ . The iron oxide content is immaterial and may be as high as 20 to 25 per cent. When used in the manufacture of aluminous chemicals and refractories it is important that the bauxite should contain as little iron oxide as possible. Bauxite for aluminum sulphate manufacture should contain a maximum of 2 per cent  $\text{Fe}_2\text{O}_3$ . Bauxite for the manufacture of refractories should preferably not contain more than 1.50 per cent  $\text{Fe}_2\text{O}_3$ . For all purposes it is important that the alumina content should be as high as possible, consistent with the price.

#### MARKETING, USES AND PRICES

*Marketing and Uses.*—As indicated, the main uses of bauxite are in the manufacture of metallic aluminum and aluminous chemicals, aluminous abrasives and aluminous refractories. In a minor way bauxite is used in the manufacture of aluminous cement, in oil filtration and in the smelting of iron. In the manufacture of metallic aluminum the bauxite is first purified by chemical methods, the resultant product, aluminum hydrate, being calcined to produce alumina. The alumina is smelted to metallic aluminum in electrolytic baths consisting essentially of molten cryolite.

The principal aluminous chemicals produced from bauxite are aluminum sulphate, aluminum hydrate, aluminum chloride and sodium aluminate. Aluminum sulphate is produced by treatment of bauxite with sulphuric acid. Aluminum hydrate and sodium aluminate result from treatment of bauxite with caustic soda. Aluminum chloride is produced by treating heated bauxite with chlorine gas.

Aluminous abrasives are produced by melting bauxite or alumina in small electric furnaces. After solidification and crystallization of this molten product, it is crushed and ground, and constitutes the crude abrasive from which grinding wheels, oilstones, abrasive cloths and papers, abrasive powders and other commercial abrasive products are manufactured.

Aluminous refractories are produced from ground bauxite or alumina by mixing them with a binder, pressing into forms and putting through a baking process. Aluminous refractories, although somewhat more

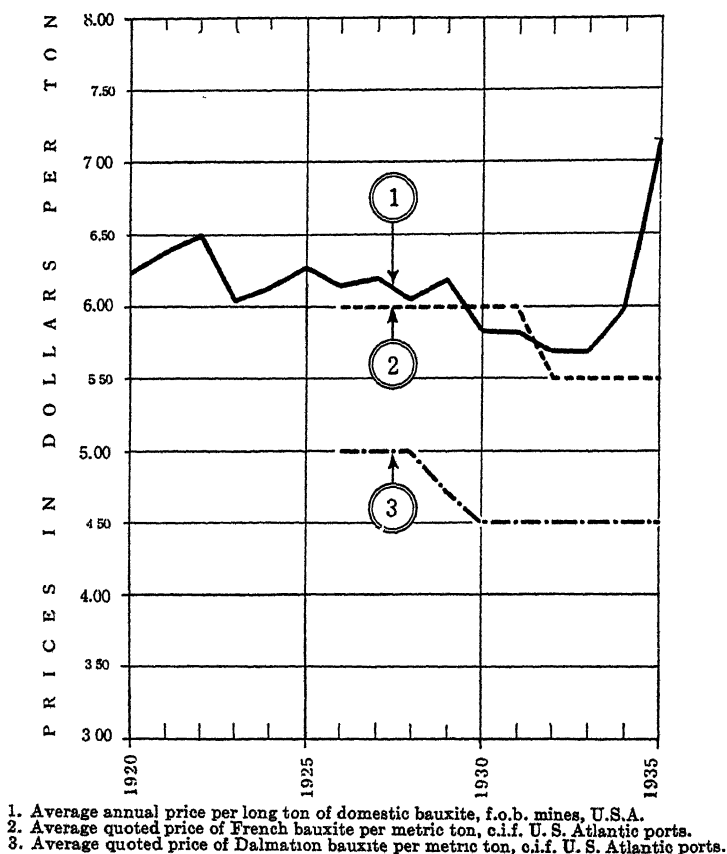


FIG. 2.—BAUXITE PRICES, 1920 TO 1935.

expensive than ordinary fire-clay refractories, are much more resistant to heat, abrasion, corrosion, spalling and erosion, and therefore are being increasingly used where severe furnace conditions are encountered. Fused aluminous refractories of the mullite type are commonly cast directly from the melt.

Aluminous cement is characterized by its rapid-hardening qualities and resistance to chemical action and heat. Aluminous cement is said

to reach the same degree of hardness and strength in 24 hr. that is reached by ordinary Portland cement in 28 days. However, there are certain super-Portland cements that harden in four or five days, and this has militated against the more extensive use of aluminous cement for general purposes.

Bauxite is said to be effective in desulphurizing, deodorizing and decolorizing oils and other liquids. It is being commercially used for this purpose in the filtering of kerosene. Other uses involving filtration are in prospect. Bauxite is used also in the smelting of iron as a slag corrective.

*Price.*—Fig. 2 shows the average price of domestic bauxite in the United States, as well as the average prices quoted for delivery at United States ports on Dalmatian and French bauxite.

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## CHAPTER VI

### BENTONITE

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**B**ENTONITES are clays that have been derived from volcanic ash and that have the clay mineral montmorillonite for their chief constituent. For many years the name was solely applied to a specific type of clay with unique characteristics, then known to occur only in the vicinity of the Black Hills of Wyoming and South Dakota. Recently, however, the term has been extended to cover a broad class of claylike materials that are petrographically similar<sup>3</sup> but that differ widely in physical properties from the original Black Hills type.

#### CLASSES

The bleaching-earth types of clays are discussed in chapter VII, therefore this paper will deal only with the technical and utilitarian aspects of the Black Hills type of bentonite.

The broad family of bentonites may be divided into two general classes: (1) those that adsorb large quantities of water, "swelling" enormously in the process, and that have the property of remaining in suspension in thin water dispersions; (2) those that absorb practically no more water than ordinary plastic clays or fuller's earths, do not swell noticeably, and settle rapidly in thin water dispersions. There are, of course, some intermediate gradations.

Type 1 has a wide variety of commercial uses, because of properties that are unique in an inorganic material. Type 2 is produced in larger tonnages, practically all of the consumption being for clarifying and bleaching in the oil industry, and is analogous to fuller's earths and bleaching clays.

Deposits of the highly absorbent Black Hills type of bentonite, described as type 1, occur chiefly in eastern Wyoming and western South Dakota, overlapping into Montana and adjacent Canadian provinces. Western Wyoming has deposits nearly similar but with slightly less absorbent properties. California contains some of this type also, although the main production of California bentonite is of the bleaching-earth variety. Utah and Nevada have scattered deposits of intermediate gradations.

\* Manager, American Colloid Co., Chicago, Ill.

<sup>3</sup> References are at the end of the paper.

The large bodies of bentonite in Texas, Arkansas, Mississippi, Kentucky and Tennessee are of the nonswelling, nonsuspendible type, as are also numerous smaller deposits in other states not mentioned here. The geological departments of some states group bentonite and fuller's earth together in their reports.

### PROPERTIES

The clay-mineral montmorillonite forms about 95 per cent of the substance of bentonite, with small fractions of feldspar, gypsum and calcite, and traces of quartz, volcanic glass, biotite and zeolites. When the raw material, without fine grinding, is dispersed in water, about 10 per cent of its particles will be larger than 10 microns in size, 15 to 20 per cent between 10 and 0.5 microns and the remainder immeasurably finer. The montmorillonite portion is in the shape of flat plates, of visible width and breadth under the ultramicroscope but of colloidal thickness, and this peculiar structure gives it a tremendous surface area.

Norton and Hodgdon,\* at the Massachusetts Institute of Technology, found some interesting facts in a series of experiments with seven finely powdered materials. Table 1 gives the computed average size of the grains of these materials down to 0.00003 cm., and the average surface area for one gram of particles.

TABLE 1

Material	Average Number of Grains per Linear Cm.	Average Area for One Gram of Material, Sq. Cm.
Dry-milled quartz . . . . .	1,400	2,900
Wet-milled quartz. . . . .	4,000	9,000
Calcined china clay... . . . .	1,100	2,400
China clay..... . . . .	2,400	5,510
Brick clay.. . . .	2,600	5,800
English ball clay..... . . . .	3,400	7,400
Bentonites <sup>a</sup> .. . . .	20,000	50,000

<sup>a</sup> Estimated from rough microscopic observation.

To get some conception of the voids between the dry particles, i.e., the dry porosity, they made a series of tests subjecting a mass of each of the dried materials to three pressures—8, 80 and 800 kg. per sq. cm. (approximately 114, 1140 and 11,400 lb. per sq. in.). Bentonite was the only one to show practically no variation in porosity. To quote, "The minimum dried spacing (of the bentonite) is about the same for all

\*Norton and Hodgdon: Some Notes on the Nature of Clay. *Jnl. Amer. Ceram. Soc.* (1932) 15, No. 3.

pressures and the porosity reached is only 6 per cent. This would indicate a close packing of a platelike material." In these tests, the bulk volume of the mass of bentonite was not reduced even at the highest pressure used.

In a further series, the materials were wetted and subjected to pressures of 8 kg. per sq. cm., and a computation was made of the water film surrounding the particles, as shown in Table 2.

TABLE 2

MATERIAL	MAXIMUM THICKNESS OF WATER FILM, CM.
Dry-milled quartz.....	0
Wet-milled quartz.....	0
Calcined china clay.....	$0.1 \times 10^{-5}$
English china clay . . . . .	$2.1 \times 10^{-5}$
Brick clay . . . . .	$3.1 \times 10^{-5}$
English ball clay. . . . .	$2.4 \times 10^{-5}$
Bentonite <sup>a</sup> .....	$3.3 \times 10^{-5}$

<sup>a</sup> Note that the bentonite has the thickest water film.

In another series the wetted masses were subjected to the same pressures applied to the dried masses. No matter what pressure was applied to the bentonite, a film of water of a certain thickness was always present and could not be squeezed out. To quote, "the thickness of the water film (on the bentonite) at any given water content cannot be reduced below a certain minimum with pressure at least up to 800 kg. per square centimeter."

A high-grade bentonite will absorb nearly five times its weight of water and at full saturation it increases in volume up to 15 times its dry bulk. With 6 to 7 parts water it becomes a gelatinous paste, the consistency of heavy grease. With 18 to 20 parts water it forms a thin sol in which the bentonite will remain in suspension indefinitely. Even in a concentration as low as one part bentonite to 5000 parts distilled water, 70 per cent of the bentonite will remain suspended for months.

The swelling property is reversible; it can be dried and re-swelled an infinite number of times and this activity is unaffected by temperatures below 450° F. However, the bentonite will not re-swell if in its first saturation cycle it is admixed with over 10 per cent of added alkali as Portland cement.

Its gels and sols increase in inherent consistency on standing, reaching their maximum in two or three days. In addition, they show pronounced thixotropic tendencies. The bentonite suspensoids are negatively charged, show Brownian movement, and are profoundly affected by electrolytes in the water. Dispersions of natural bentonite have an average pH of about 9.2, which is also their isoelectric point.

Three factors cause bentonite to swell in water: (1) water penetrates the crystal lattice of the mineral (no other crystal has this property, so far

as known) expanding the lattice by capillary pressure; (2) water forms a stable film around each particle, pushing them apart; (3) the particles mutually repulse each other because of like negative polarity.

Actually, the "swelling" is a diffusion of bentonite particles through the water; they take up more space in water than in air because they become bulkier as their crystal structure expands and because the individual envelopes of water move with the particles. Thus the volume of "free" water is reduced and the mass assumes a viscous consistency. The total volume of this mass, however, will be no greater than the sum of the original bentonite and water volumes.

### ORIGIN

The Black Hills bentonites were deposited originally as volcanic dust, which settled in the salt seas that covered the Northwest. The region alternated between land and sea and the glassy particles—of which traces are still found in the present bentonite—were gradually divitrified by chemical decomposition and hydration. The seepage of salt and alkaline waters and the dissolving of fragments of other minerals slowly altered it to the gel-forming bentonite of today.

### MINING AND PRODUCTION

The chief region of commercial production is in Wyoming and South Dakota, in a horseshoe-shaped area lying north and west of the Black Hills. A small quantity of a related but not exactly similar kind is produced in California and a very small amount in British Columbia.

The commercially workable Black Hills deposits are from 2 to 4 ft. thick, close to the surface, and contain 25 to 45 per cent moisture. Overburden from a few feet up to 10 ft. is removed, and the bentonite is dug and hauled by trucks to the processing plants, which sometimes are 10 to 15 miles away. It is dried in rotary driers down to 8 per cent moisture, then granulated or powdered for shipment. The usual commercial grade is in the form of a powder 90 to 95 per cent minus 200-mesh by dry sieve test, and is packed in 100-lb. bags.

While there was some consumption of bentonite as early as 1888, it was mainly experimental and actual production was irregular and in small quantities up to 15 years ago. In 1925 shipments were about 2500 tons from two plants and in 1935 were 42,000 tons from six producing plants in Wyoming and South Dakota.

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\* German scientists\* report the swelling of the crystal structure of bentonite as follows:

PERCENTAGE OF WATER IN BENTONITE	SWELLING, Å.
8 7	11.2
23	15.2
30	19.6

## USES

More than half the Black Hills type of bentonite produced is used in metal foundries as an ingredient of molding sands. It has bonding properties far greater than other clays, so that less clay is required in the sand, thus allowing more free space between the sand grains for rapid egress of the steam and gases created during the pouring of the metal. It also permits the sand to be worked with lower moisture, thus making less steam.

In sinking oil wells bentonite is used for thickening the drilling mud so that it will suspend rock cuttings and carry mud-weighting materials; also because of its property of sealing the walls of the well against water filtration. A new and growing use is for stopping water flow or leakage in engineering works. A thin blanket of bentonite placed on the outside of a subsurface building wall or floor or between forms, as in mine seals, trenches or cofferdams, will effectually stop the percolation of water. A slurry of bentonite pumped under pressure into subsurface sand, gravel or seamy rock strata, as under dams, reservoirs, etc., will seal the voids against water passage.

Bentonite performs detergent functions, owing to a combination of colloidal activity and high absorption (particularly for carbonaceous soil) and hence a considerable volume is consumed in laundries, especially for washing heavily soiled articles. It is used as an additive plasticizing and bonding agent in combination with other clays and ceramic materials to improve burned clay products. Many heat and sound-insulating blocks, plasters and cements employ bentonite for their bonding medium. It is a standard suspending, spreading and adhesive agent in horticultural sprays and insecticides. A recent development is the use of bentonite for clarifying turbid waters and purifying sewage.

Other established uses are: for emulsifying asphalts and other water immiscibles; as an admixture in concrete to improve workability and flow and to prevent segregation; to inhibit the gumming of screens in dewatering paper pulp; to gelatinize wet-mash poultry foods; for clarifying wines; in cosmetics and pharmaceuticals; as a suspending, thickening and paste-forming agent in a wide variety of products and processes.

## PRICES

Powdered bentonite in bags, in carload quantities f.o.b. Black Hills producing plants, sold at \$30 per ton and upwards from 1923 to 1927. In 1928 and 1929 it ranged from \$25 to \$17 per ton and has since steadily declined, selling for \$11 and \$13 per ton in 1936. In dried, coarsely crushed form, shipped in carloads in bulk, the price in 1936 was \$7 to \$8 per ton.

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## CHAPTER VII

### BLEACHING CLAY

BY J. W. BELL\* AND S. R. FUNSTEN†

THE term "bleaching clay" as used in the oil industries refers to clays that in their natural state, or after chemical activation, have capacity for adsorbing coloring matter from oil. Bleaching clays are frequently classified into two groups—those naturally active and those active after artificial activation with acid. Actually no sharp line can be drawn in this classification, since some varieties of naturally active clays can be made more active by artificial activation. Other naturally active clays are not appreciably affected by acid-treatment. Commercial chemically activated clays are generally several times more efficient than the best quality of natural commercial clays.

Fuller's earths fall within the general group of bleaching clays, and the commercial term "fuller's earth" has gradually grown to apply to natural clays, for the most part originating in southeastern United States (principally Georgia and Florida), susceptible of manufacture into granular grades for percolation. Earths of this type have not responded satisfactorily to acid activation. Fuller's earths are prepared in pulverized as well as granular form, therefore are adaptable to both the contacting and percolation processes.

The clays falling within the activable group are generally of bentonitic origin and are especially prevalent in southwestern United States. Because of the difficulties in obtaining a satisfactory granular product, these clays are prepared commercially only in the pulverized grade.

For the most part, bleaching clay is used commercially for refining oil. It is applied either by the percolation method or by the contacting method. Percolation refers to the passage of oil through a bed of granular clay, while contacting refers to direct agitation of oil and pulverized clay. In either case the refining action is obtained by intimate contacting of liquid and solid adsorbent.

#### COMPOSITION AND PROPERTIES

*Composition.*—The raw clays from which bleaching earths are prepared are composed of hydrous aluminum silicates containing varying quantities of magnesium, iron, calcium and other substances. Chemical

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composition is not a reliable means of discrimination between nonactive, active and activable clays, therefore it has not been possible to forecast the quality of any clay on the basis of the analysis alone. Careful research on some thousands of deposits of activable clay with similar analyses produced only a relatively small number of high enough quality for commercial use. Table 1 gives typical analyses of representative clays of the foregoing classifications.

TABLE 1.—*Typical Analyses*

Analyses Computed on Percentage by Weight	Nonactive Clays			Active Clays		Activable Clays	
	Siliceous Fire Clay	Florida Kaolin	High- diatom Clay	Commercial Fuller's Earth		Arizona Bentonite	
				Georgia	Florida	Activable	Activated
SiO <sub>2</sub>	72.05	45.67	71.35	53.42	52.92	50.03	55.70
Al <sub>2</sub> O <sub>3</sub>	17.21	38.45	11.45	10.06	9.42	15.00	13.13
Fe <sub>2</sub> O <sub>3</sub>	1.25	0.75	6.93	3.58	3.65	2.92	2.44
TiO <sub>2</sub>		0.10	0.47	0.52	1.18	0.84	0.33
CaO	0.45		1.79	1.29	1.91	2.60	0.31
MgO	0.75	0.05	2.00	9.18	9.08	4.54	3.46
K <sub>2</sub> O		0.06	0.24	0.64	0.98	0.12	0.13
Na <sub>2</sub> O			0.13	0.02	0.03	0.03	0.12
Cl			None	0.03	0.02	0.01	0.01
P <sub>2</sub> O <sub>5</sub>			0.57	0.12	1.21	0.02	0.05
SO <sub>3</sub>			None	0.02	0.04	0.04	0.72
CO <sub>2</sub>			Tr.	0.05	0.11	0.02	0.01
Ignition loss	8.56			9.42	10.19	8.29	7.48
Uncombined water		14.86	5.30	11.83	9.06	15.63	15.77
Totals	100.27	99.94	100.23	100.18	99.79	100.14	99.65

*Properties.*—Properties of bleaching earths from different parts of the country or even in different parts of one deposit frequently vary over a considerable range, therefore it is necessary to generalize in discussing this subject. The fuller's earth type of clay is characterized by a lack of plasticity, large water content, foliated structure, and tendency, when water is removed from the clay, to adhere strongly to the tongue. Activable and bentonitic clays differ from fuller's earth in that they are characterized in general by waxy appearance and slaking or disintegrating rapidly in water; also, activable clays do not stick to the tongue as readily as fuller's earths. The best grades of bleaching earths are light gray or brown in color in the raw, wet state and nearly white after drying. However, some earths with colors ranging from brown to buff, cream and blue are of excellent quality. Activable bentonites slake but do not swell in water as much as the almost inactivable Wyoming bentonites.

Activable clays as a rule are heavier and denser than clays such as fuller's earth. A cubic foot of dried, ground fuller's earth weighs only a little more than half as much as the same volume of dried, ground Arizona activable earth or some naturally active earths in Texas.

Bleaching earth generally occurs in clearly defined horizontal strata. The naturally active clays generally reach a thickness of 8 ft., and frequently greater than that. Clays of the activable type rarely exceed 3 to 4 ft. in thickness of strata. The clay strata of good deposits are seldom contaminated by limestone or sand.

There have been numerous publications regarding geology and mode of occurrence of bleaching earths; among them papers by Nutting,<sup>27</sup> Ross and Kerr,<sup>14</sup> T. W. Vaughan<sup>15</sup> and C. L. Baker.<sup>4</sup> The Georgia and Florida fuller's earth is thought to have been deposited as a calcareous clay in shallow Marine water. Activable earths are thought to be formed from the alteration of volcanic ash deposited as vitreous tuffs which were later devitrified.

*Theories of Behavior of Bleaching Clays.*—Bleaching clays were formerly divided into two groups widely different in properties: (1) the naturally active and (2) the activable clays. However, scores of clays having all intermediate degrees of behavior have been found and studied. A few samples showed bleaching power equal to the best fuller's earths and yet responded satisfactorily to acid activation. This gradation suggests that many fuller's earths were derived from bentonites (hydrated volcanic ash, low in silica) by natural leaching in surface water assisted in some cases by plant acids and bacteria, all of which have been found by laboratory tests to modify clays profoundly. Such an origin is further indicated by the presence of nearly pure bentonite at the bases of thick beds of some fuller's earth deposits in central Georgia and northern Florida. Ash deposited *in situ* became bentonite; that which gradually washed in, with leaching of grain surfaces, became fuller's earth, sometimes very free from silt.

After room-drying, lumps of bentonite slake readily in water; lumps of fuller's earth do not, or at most crack into large chips. The northern bentonites (Wyoming) swell and gel on slaking.

Samples of fuller's earth, on room-drying, change very little in bleaching power, usually improving slightly with the dehydration.

#### DISTRIBUTION OF DEPOSITS

Bleaching-clay deposits are well distributed throughout the world, but more clays and larger deposits have been found in the United States than elsewhere. Countries known to produce bleaching earths for clay consumption are: United States, England, Germany, Russia, France and Japan. Clays susceptible to activation to a high degree have been found

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<sup>27</sup> References are at the end of the chapter.

in Germany and France and in the states of Arizona, Mississippi, California, Oklahoma, Texas, Florida, Georgia, Arkansas, Colorado and Utah. While natural bleaching earths are widely produced and distributed, there are relatively few places in the world where commercial activated clays are produced. United States and Germany are leaders in the world production of activated clay. A considerable quantity of the German product is used in the United States. Germany's natural advantages have been lower rates for labor, lower cost for acid used in the process, and extremely low freight rates to the United States. A protective tariff tends to equalize the cost of the German with that of the domestic product. Substantial quantities of natural clays are imported from England. However, here again the domestic industry is protected to some extent by the tariff. The known deposits of bleaching earth in the United States are more than sufficient to meet all conceivable requirements for years to come.

#### PRODUCTION AND CONSUMPTION

Bleaching earth has been used in various ways for many centuries. Mention is made of it in the Bible and in the writings of Pliny. In modern times England has mined fuller's earth, and up to the twentieth century was practically the only source of the material. Fig. 1 gives the

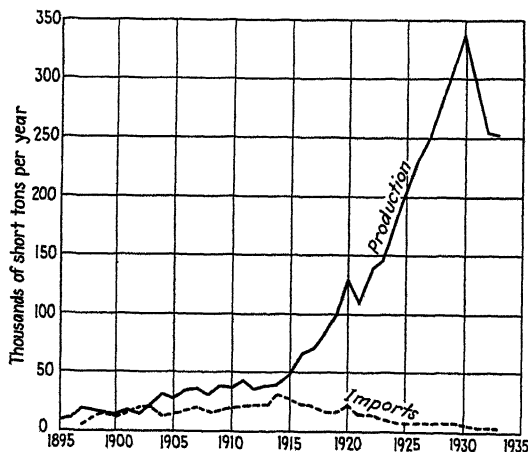


FIG. 1.—TRENDS IN PRODUCTION AND IMPORTS PER SHORT TON OF FULLER'S EARTH, 1895-1935.

After W. W. Adams and R. W. Metcalf: Fuller's Earth. Minerals Yearbook, 1934.

trends in production and imports per short ton of earth during the period 1895 to 1933. This chart indicates production of all naturally active earths, including fuller's earth but excluding activated earths. American production has grown rapidly in recent years, although for certain uses English fuller's earth is still brought into the United States.

Activated clay was manufactured first in Germany about 30 years ago, and in this country some 15 years later. The petroleum refiners were quick to see its value and now production has increased to the point where the United States meets most of its own requirements. The total world production of activated clay at the present time is believed to exceed 50,000 tons annually, about half of which is produced in the United States. As stocks are usually small, consumption corresponds closely with production.

Prior to the twentieth century bleaching clay was used chiefly for treating vegetable and animal oils. At present more than 90 per cent of the entire production is consumed in the petroleum industry. The consumption of activated clays has been greatest during the last decade. This type has been increasingly in demand on account of its high efficiency compared with naturally active clays. Even during the last five years, when there has been no increase in the use of other bleaching clays, the use of activated clays has more than doubled.

#### PROSPECTING, EXPLORATION AND MINING

*Prospecting and Exploration.*—The United States Geological Survey has made a rather extensive study of bleaching earths in the United States and various publications give details as to the location and nature of deposits covered in these studies. The relatively low value of bleaching earth makes it of little interest to the individual prospector. Most of the large companies do this work as needed. Many samples are received by these companies from landowners, and if the tests show promising results the deposits are inspected and samples obtained by open-cut methods or by use of hand core drills. Since the quantity of naturally active and activable clays in the United States is far in excess of any conceivable demand, prospecting and exploration are limited to properties for which location and mining conditions are particularly favorable.

*Mining Methods.*—Mining of bleaching earths for the most part is by the open-cut method. The overburden, which ranges from 5 to 50 ft. in depth, is removed either by steam shovel or dragline to within approximately one foot of the top of the clay. The remaining foot of overburden is removed carefully by hand or mechanical means. It is the usual practice to lay out the area carefully before beginning work, so as to evaluate fully the quality and amount of material to be mined. Whenever possible, mining is done in dry weather, in order to avoid excess handling of moisture. When the clay strata are too deep for open-cut mining, a tunnel method is used, very similar to that used in coal mining.

Great care is taken in mining to eliminate foreign material such as limestone or other rock. The water content of clay as mined depends largely upon the location and type of clay. The amount of uncombined water in southeastern clays as mined is approximately 50 per cent by

weight, while in the Arizona and California clays it varies from 20 to 30 per cent by weight. In some instances the lump clay is hauled from the mine to the mill in mine dump cars, but when the distance is too great the material is generally transferred by truck and rail. When weather conditions permit, much of the water is removed from the clay by air-drying before it is transferred.

### PREPARATION FOR MARKET

#### FULLER'S EARTH

When the bleaching earth is to be used in the natural state without activation or granulation, the preparation for market is comparatively simple. It is only necessary to pass raw lump clay through a crusher, dry the crushed clay by means of a gas, wood or oil-fired rotary drier, and finally to grind the dried clay to the desired size. Dust losses are insignificant, because little money is spent on the material. It is important to

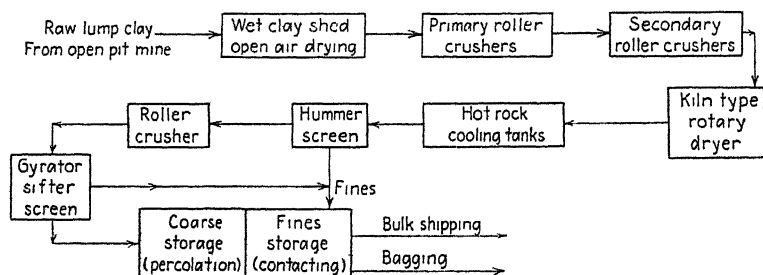


FIG. 2.—FLOWSHEET OF FULLER'S EARTH MANUFACTURE (PERCOLATION GRADE).

maintain careful technical control of temperature and time in the drying operation, because the clay can be injured greatly by incorrect handling. Experience has shown that a free moisture content of from 8 to 10 per cent gives the greatest efficiency for natural clays of the contact type. This corresponds approximately to a total volatile content of 15 per cent. The preparation of granular fuller's earth is somewhat more complicated.

The procedure in a typical fuller's earth plant in the United States is as follows: After the raw clay has been air-dried for two or three days, it is loaded into two portable distributors, each equipped with a hopper bottom and a steel-plate apron conveyor. The clay is fed at a uniform rate by the distributors onto a 24-in. inclined belt conveyor that discharges into two or more roll crushers which reduce the clay to a maximum size of 2 in. From the primary roll crushers it passes to bucket elevators that discharge it to five secondary crushers, which reduce it to a maximum size of 1 in. Oil-fired rotary driers housed in an adjoining building follow each set of crushers. The clay, in passing through a drier, is lifted by longitudinal interior channel irons and dropped through

hot gases. In the 20 minutes required for the clay to pass through a drier, its volatile content, including moisture, is reduced from 40 or 50 per cent to a maximum of 15 per cent. Chain drag conveyers of the bucket type move the product of the driers to three clay-cooling tanks which are well ventilated to carry off suspended dust, heat and moisture. By means of chain bucket elevators, the clay discharges into the top of the two tanks of larger diameter. Through a series of feed-regulating gate valves in the bottom of these tanks, it passes onto a conveyer belt, discharging into the top of the third tank, which has a hopper bottom and a mechanical feeder through which a uniform quantity of clay is fed to a  $\frac{1}{2}$ -in. mesh, electrically vibrated Hummer screen. The oversize, after being reduced in a high-speed roller mill, joins the undersize and passes directly to a belt conveyer equipped with a magnetic head pulley, which removes all tramp iron. The clay is then taken to the top of the mill building by a bucket elevator of the belt type, and passes in two equal streams on each side of a fire wall that is built on the center line of the building. Operations on each side of the wall are identical but distinctly separate from each other. The initial separation in the mill building is made by a Hummer screen of the same type employed under the clay-cooling tank. Reduction is effected in roller mills, which consist of duplicate sets of corrugated rolls. From each set of rolls the material is conducted to a silk-dressed gyrating sifter. Handmade silk of Swiss manufacture is used in these sifters. It is exceptionally uniform in quality, is durable and can be repaired readily. The oversize, or tailing, from each sifter passes directly to another set of rolls for further reduction and screening. This operation continues until the material has been reduced to the required mesh. After screening, the clay passes to a series of hopper-bottom bins, in which the various sizes are stored. Shipments are made in bags and in bulk.

For bag shipment the clay is weighed automatically and filled into bags of the best quality of burlap, usually 135 lb. to a bag. The bags are sewed shut, tagged, transported by hand truck to the shipping platform and placed in cars with the long dimension horizontal. Carloads consist of 25 to 35 tons. Bags are the property of the producer, and are credited on the consumer's account when they are returned in good condition. Milling practice in general is similar to that employed in milling wheat. The object of gradual reduction is to limit as much as possible the percentage of fines, the principal market demand being for the coarser or granular grades. Grain-type freight cars are used for both bag and bulk shipments.

#### ACTIVATED CLAY

The manufacture of an activated clay is more complicated than the manufacture of the granular fuller's earths (Fig. 3). There are seven

important steps in the process: (1) preparing the raw clay for charging to treaters or reaction vessels; (2) activating; (3) washing out impurities; (4) dewatering mechanically; (5) drying; (6) grinding to proper mesh size; (7) packaging.

The raw clay for charging to treaters may be prepared by either wet or dry method. When the wet method is employed water is added to the raw clay, which is already high in moisture, until it forms a slurry or thick slime, which is then conveyed to treaters by pumps or gravity. When the so-called dry method is employed, much of the free moisture is removed from the clay by a predrier. The dried clay is then usually conveyed by belts to a mixer, where water is added in order to make a slurry as it enters the treaters. Different methods are employed, depending upon the nature of the clay. Sometimes, instead of using ordinary water for making a slurry, a "sour" water or return liquor from the washing system is used. The clay and water, or liquor, are accurately measured before

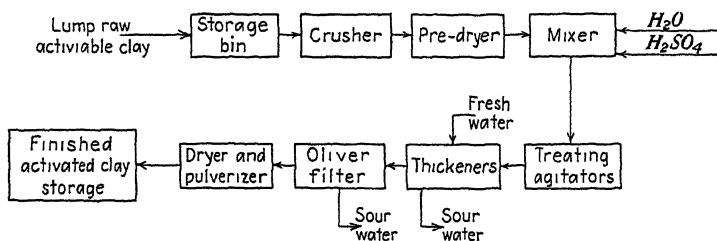


FIG. 3.—FLOWSHEET OF ACTIVATED CLAY PROCESS.

going to the treaters. The acids used for the activation are usually sulphuric or hydrochloric, generally sulphuric in this country, because of its lower cost. The acid is usually added to the slurry as it enters the treaters, although sometimes it is added during mixing or after the charge has entered the treaters. The proportion of acid varies according to its concentration and the nature of the clay. Steam is used to bring the charge to boiling point, and during the treatment the temperature is kept between 212° and 220° F. The charge is kept in constant motion during treatment by means of air or mechanical agitation. The length of time of treatment varies with the nature of the clay and the quantity and concentration of the acid; it is usually between 2 and 12 hours.

The so-called continuous method of treatment is sometimes employed. In this a proper mixture of the clay, water and acid moves in the form of a slurry from one treater to another in a series, as many treaters being used as are required to give the proper time for activation. All treaters must be made of or lined with corrosion-resisting materials.

After the activation has been completed, the slurry is conveyed to a countercurrent thickener system, where the water-soluble constituents are removed from the clay. A thickener system consists of a number of tanks

connected in series, one elevated slightly above the other. In each tank there is a shaft to which are attached at the bottom a number of rakes, usually four. The shaft is suspended in the center of the thickener and the rakes reach nearly to the outer edge. The speed of revolution of the shaft varies according to the settling rate of the solids. On the bottom side of the rakes are numerous blades set diagonally. As the solids settle they are dragged by the blades slowly to the center of the thickener, where they drop into a cone from which they are conveyed through a pipe line by means of a diaphragm pump, and discharged into the thickener next higher in the series. This operation is repeated through all thickeners of the system. At the same time there is uniform flow of fresh, soft, hot water entering the highest thickener of the system and moving down through the entire series, carrying with it a large part of the free acid, dissolved salts, and other impurities that have been separated from the solids. Sometimes the washing is done by means of filters and fresh water in a batch system and sometimes by filters connected in series in a continuous system. All pumps and lines used for conveying concentrated slurries must be of corrosion-resisting materials. For this purpose Duriron and lead have been very satisfactory.

From the thickener system the slurry is transferred either by pump or gravity to a continuous vacuum filter. The effluent is pumped away for further use in washing the slurry, or is discarded. The cake from the filter is conveyed to a drier, where its uncombined moisture is reduced to approximately 15 per cent. The dried material goes to a grinder and is pulverized to the size desired. Sometimes the drying and grinding are done in the same equipment. After the material has been ground it is usually packed by bagging machines into multiwall paper bags. Some bags are of the self-closing valve type, and others are of the open mouth type. When the latter are used the charge is weighed in the bagging machine and dropped into the bag and the closing is done by a sewing machine, which is part of the equipment. Multiwall paper bags have been used for activated clay during the entire life of the industry in the United States. They are used for shipments to all continents and are accepted by all classes of carriers.

Technical control of plant operations is essential at each stage in the process, in order to ensure a uniformly high quality of the final product.

#### TESTS AND SPECIFICATIONS

The specifications for bleaching earths depend largely on the nature of the work to be done. For refining vegetable, animal and mineral oils by the contacting method, a finely pulverized clay is used, having a mesh of at least 50 per cent through 200 and generally 90 to 95 per cent through 200. The limiting factor on fineness is filter rate, or the speed with which the oil can be separated from the clay cake after contacting. If too much



impalpable powder is present, the filter rate may be retarded to a point unsuitable for refiner's use. Therefore, while it is an advantage from the standpoint of rapidity of decolorizing action to employ fine-mesh clay, the degree of fineness must be held within definite limitations. When the percolation method is used a coarse mesh is required in order to permit free passage of the oil through the clay. The most popular percolation grade is that known as 30/60 mesh. Other standard-mesh percolation grades are 60/90 and 15/30. Aside from meshing, the specifications for naturally active clays are very similar, whether they are contacting or percolation grade.

Table 2 gives typical physical tests of a naturally active commercial southeastern fuller's earth of fine and coarse grades, of western natural clay, and of fine-grade artificially activated clay. This table refers to physical tests only because the most important specification for a bleaching earth is its decolorizing value. A list of tests commonly applied in the bleaching-earth industry follow, together with a brief outline of each test:

TABLE 2.—*Typical Tests on Representative Clays*

Dry Natural Clay	Fuller's Earth, Florida-Georgia Type		Natural Bleaching Clay, Southwest Type, Fine Grade	Artificially Activated Clay, Domestic Type, Fine Grade
	Fine Grade (Contacting)	Coarse Grade (Percolation)		
Volatile matter, per cent	16.0	16.5	16.0	21.0
Density, lb. per cu. ft. . . . .	31 0	35.0	53.0	36 0
Acidity, mg. KOH per gram . . .	Neutral	Neutral	Neutral	3 5
Screen test, per cent through 200 mesh . . . . .	95 0		70.0	88 0
Mineral-oil decolorization value (efficiency $\alpha$ = 100 per cent), per cent . . . . .	100		40-110	250-500

A. PHYSICAL AND CHEMICAL INSPECTION TESTS APPLICABLE TO BOTH NATURAL AND ARTIFICIALLY ACTIVATED CLAYS.

**Volatile Content.**—The percentage loss in weight of a given sample of clay after ignition at 1800° F. for 10 minutes is known as the per cent volatile matter of the clay.

**Moisture Content.**—The percentage loss in weight of a given clay sample upon drying to constant weight at 220° F. is known as the moisture content of the clay.

**Volume Weight of Clay.**—The volume weight of bleaching-clay samples is determined by observing the volume occupied by a given weight of clay after five minutes mechanical tamping time, in a graduated cylinder, the results being expressed in pounds per cubic foot.

**Screen Analysis.**—A quantitative screen test is made on a given bleaching-clay sample by determining the weight per cent of clay retained by and passing through each of a series of standard measuring screens using the conventional Ro-Tap.

- Acidity.—The bleaching-clay acidity comprises the weight of potassium hydroxide in milligrams required to neutralize a distilled water extract from one gram of clay, phenolphthalein being used as an indicator.
- Oil Retention (contact grade clays only).—Oil retention characteristics of fine-mesh bleaching clays are determined by measuring the increase in weight of a given quantity of clay after contacting with oil and blowing the cake produced with air at 40 lb. per sq. in. pressure at a temperature of 375° F. Correction is made for any change in water content when calculation is made for oil retention.
- B. ACTIVATION METHODS.—Natural bleaching clays, exhibiting potential activable characteristics, are activated by treatment in weak acid (i.e., 8% to 20%) at boiling temperature for six hours, washing the residual slurry free of acid and soluble salts, drying to 20 per cent volatile content and pulverizing the treated dry product.
- C. DECOLORIZATION TESTS.
- Percolation-grade Clays.—The decolorizing power of percolation-grade clay is determined by permitting a standard oil stock to pass through a column of granular bleaching clay and measuring the volume of filtrate obtained at a given composite filtrate color. By comparison of this yield with the volume of filtrate obtained under identical conditions through the use of a clay of known decolorizing power, it is possible to determine the efficiency of the unknown clay as compared with the standard.
- Contact-grade Clays.—The decolorizing power of the contact-grade clays is gauged by determining the color produced by mixing the clay with a standard oil stock, agitating the mixture at a given temperature, and filtering the clay from the oil. Knowing the amount of a standard clay required to produce the same color, the decolorizing power of the clay being treated can then be expressed as per cent of the standard.

Since there is no single oil standard by which efficiency of the various earths are gauged, the relative values of competitive clays must be established on separate customers' stocks. The importance of oil-retention value of the competitive earths is second only to decolorization value, since the oil held up or absorbed in the clay after use is not customarily recovered and therefore the loss must be charged off to the operation when the clay is discarded. This particular test is not so important with the percolation-type clay, because revivification is generally practiced, but it is of utmost importance for contacting clays, where oil is frequently not recovered from the spent cake. A great advantage for chemically activated clays is the low oil retention, owing to the fact that only a small quantity of material is required to obtain decolorization.

#### MARKETING, USES AND PRICE

Fig. 4 shows a typical contact filtration plant for processing mineral oils with bleaching earth. This plant is semicontinuous and differs from the plants for bleaching vegetable and animal oils, which are generally batch operations. Fine-grade clay (100 mesh and up) is used exclusively in contact filtration plants. A typical percolation process for refining mineral oils is shown in Fig. 5. Among the principle types of oil refined

in the mineral-oil field are naphthas, fuel oils, lubricating oils, waxes and greases. The more important vegetable and animal oils to which bleaching clays are applied are cotton seed, soya bean, linseed, lard, coconut, palm and tallow.

The principal object of applying bleaching earths is to remove color, but there are increasingly important uses in all fields of refining that do not involve color removal. For instance, in treatment of naphtha gum

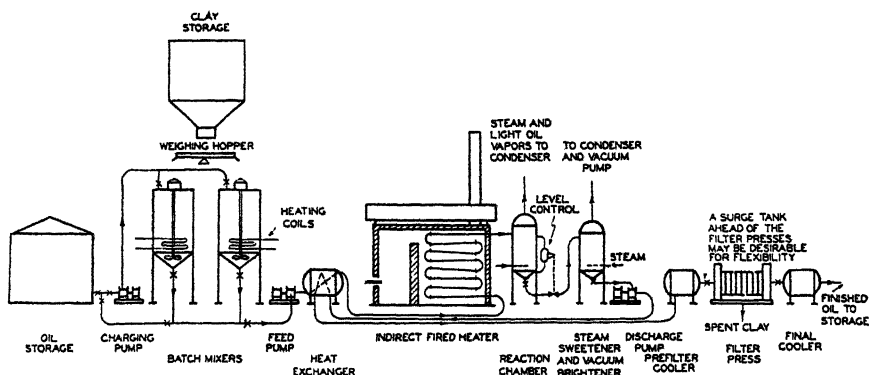


FIG. 4.—FLOW DIAGRAM FOR CONTACT FILTRATION PLANT.

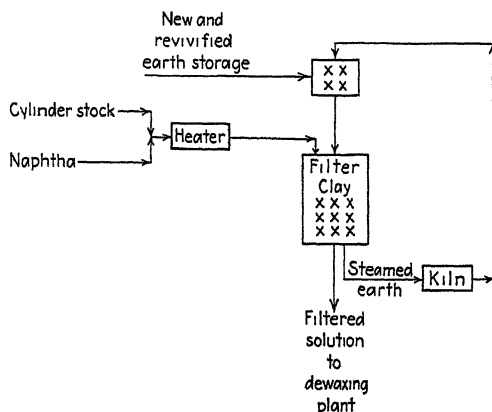


FIG. 5.—DIAGRAM OF MINERAL OIL PERCOLATION PROCESS (CYLINDER STOCK SOLUTION).

reduction and stability are more important factors than removal of color. In refining of lubricating oil improved sludge content, carbon content, oxidation test, acidity, emulsion test, viscosity index, etc., are often more important, and in the vegetable-oil field stabilization and removal of odor and taste are often the more important factors.

Some of the results secured with bleaching earths can be accomplished in part by other means of refining, such as solvent treating, acid treating, hydrogenation or fractionation, but the relatively low cost of the bleaching-earth process makes it extremely advantageous.

Deliveries of bleaching earth are made both in box and tank cars, but for the most part the clay is shipped in paper or burlap bags. On account of the wide variation in uses and type of equipment employed throughout the country, producers of bleaching earth maintain adequate staffs of experienced engineers to offer technical service to the customer.

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## CHAPTER VIII

### BORAX AND BORATES

BY WALDEMAR T. SCHALLER,\* MEMBER A.I.M.E.

**B**ORAX and boric acid are the two principal compounds of boron that are of commercial interest. Borax occurs in nature in an impure form. Formerly it was extensively manufactured from the lime borates, colemanite and ulexite, which are not soluble in water and require treatment with a solution of sodium carbonate to produce refined borax. The sodium borates, borax and kernite, however, are soluble in water and lend themselves readily to treatment.

The properties that render borax desirable and useful are its water solubility, low melting point, and fluxing properties. Its usefulness in these respects extends into many distinct fields.

Boric acid does not occur independently in nature in large quantities but is manufactured from borate minerals and salts. It is also obtained by condensation from natural steam in certain parts of Italy.

The development of borate mining in the United States has been featured by the successive discarding of one mineral for another that could be used more effectively. At first (1864-1872) native borax, or tincal, was obtained from lake muds. This source of supply was soon displaced (1872-1887) by ulexite and borax from the playas and borax marshes. As these deposits were of relatively low grade and erratic in their mode of occurrence, they were displaced (about 1887) by bedded deposits of colemanite in larger and purer bodies, together with associated massive ulexite. The later (1925) discovery of high-grade deposits of borax and kernite and the successful extraction of borax from the brine of Searles Lake again changed the source picture, and these sodium borates are now the chief source of supply for refined borax.

#### COMPOSITION AND PROPERTIES

While the complete list of minerals containing boron is long—about 60 are known—only about half a dozen have been of much commercial importance. The important borate minerals in the United States that have served as source material for the preparation of refined compounds of

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boron are listed in Table 1. About two-thirds of all these important borate minerals, when calcined, consist of anhydrous boric acid ( $B_2O_3$ ).

TABLE 1.—*Important Borate Minerals of the United States*

Name	Formula	Percentage of Anhydrous Boric Acid ( $B_2O_3$ )	
		In Mineral	In Calcined Mineral
Borax ..	$Na_2O.2B_2O_3.10H_2O$	36.6	69 2
Kernite... . . . .	$Na_2O.2B_2O_3.4H_2O$	51.0	69 2
Colemanite . . . . .	$2CaO.3B_2O_3.5H_2O$	50 9	65 2
Ulexite. . . . .	$Na_2O.2CaO.5B_2O_3.16H_2O$	48.0	66 6

Other borate minerals in the United States but of minor commercial value, and usually associated with those named in Table 1, are:

Tincalconite. . . . .	$Na_2O.2B_2O_3.5H_2O$
Probertite .. . . .	$Na_2O.2CaO.5B_2O_3.10H_2O$
Meyerhofferite . . . . .	$2CaO.3B_2O_3.7H_2O$
Inyoite . . . . .	$2CaO.3B_2O_3.13H_2O$
Priceite .. . . .	$5CaO.6B_2O_3.9H_2O$
Bakerite. . . . .	$8CaO.5B_2O_3.6SiO_2.6H_2O$
Howlite. . . . .	$4CaO.5B_2O_3.2SiO_2.5H_2O$

In foreign countries, sassolite ( $B_2O_3.3H_2O$ ) and boracite ( $5MgO.MgCl_2.7B_2O_3$ ) have also been utilized commercially.

The term borax has been used loosely for almost any borate. The following synonyms have been used in the trade and in the literature instead of the correct and accepted names:

ACCEPTED NAME	INCORRECT SYNONYM
Borax. . . . .	Tincal, prismatic borax, sodium baborate, sodium tetraborate
Tincalconite. . . . .	Octahedral borax, mohaveite
Kernite. . . . .	Rasorite
Ulexite . . . . .	Boronatrocaltite, natroborocalcite, hayesine, franklandite, cryptomorphite, tiza, tinkalzitite, borate of lime, cotton-ball, sheet cotton
Probertite .. . . .	Boydite, kramerite
Priceite.... . . .	Pandermite
Howlite .....	Pandermite
Boracite.....	Stassfurtite

The borate minerals chiefly produced in the different countries are as follows:

United States ... . . . .	Borax, ulexite, colemanite, kernite
Chile (also Argentina, Bolivia and Peru) Ulexite	
Turkey.....	Priceite

Italy....	Sassolite
Germany..	Boracite
Tibet.....	Borax

Borax is found in individual crystals, as aggregates of poorly developed crystals, and as compact glassy masses showing no crystal faces. When pure, borax is clear and colorless but much of it is dull earthy white, because of partial dehydration to the lower hydrate tincalconite, and may be gray or dark colored opaque owing to enclosed mud. The crystals are monoclinic and have several cleavages, but the natural mineral seldom shows cleavage faces when broken. Its hardness is 2 to 2.5 and its specific gravity is 1.72. Borax is soluble in cold water but much more so in hot water. It swells and fuses when heated.

Kernite occurs in clear, colorless monoclinic crystals as much as several feet in diameter and in cleavable masses of similar size. Two perfect cleavages and several other planes of separation cause the mineral to break readily into small pieces and fibrous aggregates. The mineral somewhat resembles selenite gypsum in general appearance. The hardness is 2.5 and specific gravity is 1.95. Kernite is very slowly soluble in cold water but readily so in hot water. It expands when heated but does not swell as much as borax.

Colemanite occurs in shining crystals, colorless to white, and also as white to gray compact granular masses. Its hardness is 4 to 4.5 and specific gravity is 2.42. The mineral is insoluble in water but soluble in acids. When heated it decrepitates to a powder, a characteristic property.

Ulexite is not found in distinct crystals but forms either rounded aggregates of radiating acicular fibers (cotton balls) or solid masses of parallel or radiating white fibers with a silky luster. The hardness is 2 and specific gravity is 1.96 (not 1.65 as given in the textbooks). Ulexite is not soluble in cold water but is partly attacked by hot water and is readily soluble in acids.

#### ORIGIN AND MODE OF OCCURRENCE

The borate minerals described occur in sedimentary beds of different kinds, mostly clay and shale largely derived from volcanic ash, and associated with limestone and sandstone. To a large extent borax and ulexite result from the repeated evaporation of intermittent shallow lakes or playas which have received the saline-rich drainage from considerable areas. These deposits, including the well-known borax marshes, began to accumulate in Tertiary time and the mineral matter in them has been augmented in places by the boron-containing products from solfataras and hot springs, which represent the last stages of Tertiary and Quaternary volcanic activity.



The kernite of the underground deposits of the Kramer district in California is the product of recrystallization of earlier beds of borax. Its smaller content of water (26.3 per cent, as contrasted with 47.2 per cent in borax) indicates that it formed at a higher temperature than borax.

The lime borate colemanite occurs as geodes, nodules, small lenslike masses, bunched lenticular layers and compact beds of massive mineral. In many localities, colemanite was formed from ulexite by leaching. The leachings, containing soluble sodium borate, became a part of the general drainage, which in favorable places may have carried this material into lakes where subsequent evaporation produced beds of borax.

The boron originally came from solfataras and hot springs connected with past volcanic activity of the region where the borate minerals are now found. In whatever combination the boron first came to the surface, it has undergone many chemical and mineralogical transformations before final fixation in the bodies of borate minerals now found. Much of the boron was probably in the form of boric acid and first became fixed as ulexite ( $\text{Na}_2\text{O} \cdot 2\text{CaO} \cdot 5\text{B}_2\text{O}_3 \cdot 16\text{H}_2\text{O}$ ) by the reaction of boric acid with the lime and soda derived from volcanic ash and clays resulting from the decomposition of the ash.

Later crustal movements have folded and crushed many of the borate beds and turned some of them on end. Different geological and mineralogical processes have altered the original structure of the sedimentary beds. Conditions have thus been favorable for a long continued series of mineralogical changes: first, the fixation of a boron mineral such as ulexite; next, leaching to form colemanite with release of water-soluble sodium borate. Borax with  $10\text{H}_2\text{O}$  changed to kernite with  $4\text{H}_2\text{O}$ , with later hydration to tincalconite with  $5\text{H}_2\text{O}$  and back again to borax with  $10\text{H}_2\text{O}$ . The lime borates form a similar series with changing water content, thus inyoite has  $13\text{H}_2\text{O}$ , meyerhofferite  $7\text{H}_2\text{O}$ , and colemanite  $5\text{H}_2\text{O}$ . The soda lime borates behave in the same way: ulexite has  $16\text{H}_2\text{O}$  and probertite  $10\text{H}_2\text{O}$ . Then come pseudomorphs of colemanite and meyerhofferite after inyoite, and of calcite after colemanite.

In all the known deposits of borax except the one at Kramer, Calif., other saline minerals, chiefly the chlorides, carbonates and sulphates of soda and of lime, together with other salts, are abundantly present. This is to be expected where such deposits are concentrations of salts brought in by drainage waters, augmented perhaps by evaporation products of boron-containing hot spring waters, which also contain similar salts in addition to borates. Searles Lake, with its wealth of chloride, carbonate, and sulphate saline minerals, is a typical example of such an association of borates with nonborate saline minerals.

The occurrence of borates of soda (borax and kernite) at Kramer, without the usual borate-free salines, therefore calls for an unusual expla-

nation. It is believed that the Kramer deposits of borax and kernite are neither simply the direct natural evaporation residues dissolved in and accumulated by surface or underground drainage waters nor are they direct deposits of hot-springs origin, but that they represent instead the concentration, evaporation, and in part, later recrystallization products, of the sodium borate leachings of ulexite, which forms in clay essentially free from the other much more soluble saline minerals named. Colemanite, formed by the leaching of ulexite, occurs abundantly in the western part of the Kramer area, where it was formerly mined. The first mineral crystallizing out of the sodium borate leachings was borax. Part of this was later recrystallized into the lower hydrate kernite, when the deposits were buried and subjected to increased temperature and pressure. Since the mines have been opened, part of the exposed kernite has changed back to borax by hydration.

The Kramer borate area in the western part of the Mojave Desert lies in southeastern Kern County, California, about halfway between Barstow and Mojave, and about 7 miles northwest of Kramer. The oval area in which the borate minerals (borax, kernite, ulexite, probertite and colemanite) have been found is 4 miles long in an east-west direction and 1 mile wide. Ulexite and colemanite seem to underlie the entire area but the sodium borates have been found only at three places in the central and eastern parts. Three mines in these places have produced borax and in two of them much kernite is associated with the borax. The beds containing the borate minerals occur in clays and shales in a synclinal basin of bedded Tertiary sediments, chiefly volcanic tuffs but with some limestone, chert, arkose, etc., resting on older granitic and metamorphic rocks. All the deposits are underground, mostly at depths between 325 and 900 ft. Alluvium covers much of the surface. It seems a curious irony of fate that the 20-mule teams, formerly trudging their weary way with their loads of borates from Death Valley to Mojave, passed almost over these immensely more valuable buried deposits. The old road lies only a few miles to the north.

No figures can be given as to the size of these deposits near Kramer, but in a report<sup>20</sup> published in 1929 the borate body in one of the mines is described as being about 100 ft. thick and extending at least 1500 ft. north and south and about 1200 ft. east and west. The production of crude borates from the Kramer area prior to 1935 is estimated as close to a million short tons. The Kramer area is capable of supplying enormous quantities of borates for many years.

Searles Lake, in the northern part of the Mojave Desert, is a roughly circular area of about 12 square miles, representing the accumulation of saline salts left by the gradual evaporation of a formerly much larger

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<sup>20</sup> References are at the end of the chapter.

body of water. It is now a flat, white porous aggregate composed chiefly of crystalline salts of sodium and potassium permeated by a dense alkaline brine, and only during the winter does the brine sometimes rise a few inches above the surface. The total solids in the brine are about 36 per cent, as follows: sodium chloride 16, sodium sulphate 7, sodium carbonate 5, potassium chloride 5 and sodium borate 3.

#### DISTRIBUTION OF DEPOSITS

The distribution of the deposits of borate minerals in the United States that have been worked is intimately related to the changing history of the development of the industry. This change is in large part due to the successive discovery and adaptation of different source materials (natural borax-ulexite-colemanite-brine-kernite and borax) used in the preparation of refined borax and other boron compounds. Therefore a brief historical outline is combined with that of the distribution of the deposits.

All the known or potentially commercial deposits of borate minerals in the United States are in the three western states, California, Nevada and Oregon. Priceite, a lime borate from Curry County, Oregon, and borax from Harney County, Oregon, have affected the borax industry but little. Total production from these deposits, just north of the southern boundary of Oregon, has been very small. California and Nevada therefore are the essential borate mineral producing states.

The first borax obtained in California (in 1856) was made by the evaporation of water from Tuscan Springs (later referred to as Lick Springs), 8 miles east of Red Bluff, Tehama County. Sodium borate was also found in solution in a number of springs in near-by counties. Very soon after the first discovery, crystals of borax were found in the muds of Borax Lake, in Lake County and the borax obtained there supplied much of that consumed in the United States from 1864 to 1868. For the next five years borax was recovered from the water and mud of near-by Hachinhama Lake. The discovery of large quantities of borax and ulexite in western Nevada and southern California in the early seventies brought to an end the first period of domestic production of borates.

Borax is reported to have first been found at Searles Lake, California, in 1862, although extraction from the surface efflorescences and underlying muds did not occur until about 1874. Borax is said to have been found in the flat basin of Death Valley in 1873, but commercial extraction began only about 1880.

In Nevada, Columbus Marsh was located in 1864 as a salt bed and the presence of ulexite was noted. In 1869 more "cotton balls" of ulexite were picked up and in the early seventies additional localities were found. Much activity in borax centered around Columbus Marsh in 1872-1873,

and the well-known marshes, Columbus, Teal, Rhodes, Fish Lake, and others, as well as Kane Springs in Kern County, Saline Valley in Inyo County, and other marshes in southern California, furnished cotton-ball ulexite and borax. In 1872-1874, carloads of ulexite from the Nevada marshes were shipped to Lake County, California, where it was treated with the sodium carbonate from Lake Hachinhama to form borax.

During the seventies and early eighties borax and ulexite from the lake muds of Nevada and California were the chief source of refined borax and this second period may be described as that of the borax marshes.

The discovery of colemanite as a new mineral in Death Valley in 1882 and in the Calico Mountains, San Bernardino County, the following year, ushered in the third period. For about 40 years, colemanite and associated ulexite from these and various localities in Inyo, Kern, Ventura, Los Angeles and San Bernardino Counties, and Clark County, Nevada, supplied much of the source material for the preparation of refined borax and other boron compounds. The mining of colemanite in massive beds many feet thick soon stopped the production of borax and cotton-ball ulexite from the surface efflorescences and muds of the marshes and dry lakes of the "borax lakes" and these were abandoned as source material about 1887.

Colemanite mining began in the Calico district, northeast of Daggett, about 1887, and the value of these deposits led to much prospecting and to the discovery of the massive commercial beds in the Death Valley region and elsewhere. Points of production changed as new deposits were opened. Colemanite was found near Stauffer, northeastern Ventura County, in 1898 and production ran continuously to 1907. The Calico district closed about 1907 and operations shifted to Ryan and Mount Blanco in the Death Valley region, to Lang in Los Angeles County, and to the Kramer district, Kern County, where colemanite and ulexite were discovered underground in 1913. The buried deposits of borax and kernite, about two miles to the east, were not discovered until 1925. Meanwhile operations in colemanite again shifted to near Shoshone, southeast of Death Valley, and to two deposits (White Basin and Callville Wash) in Clark County, southern Nevada. During this third or colemanite period, considerable quantities of ulexite were imported from Chile and to a lesser extent from Argentina, Bolivia and Peru.

The beginning of production of refined borax from the brine of Searles Lake in 1920 and the discovery of immense deposits of sodium borates (kernite and borax) in the Kramer district in 1925 soon closed the mines of the lime borates and the colemanite period saw its end. Today the Kramer and Searles Lake deposits of sodium borates furnish, and for some

time to come are likely to furnish, almost the entire supply of borax and other boron compounds for the world. The present (1934 and 1935) production of nearly a quarter of a million tons annually of borates from near Kramer and at Searles Lake has relegated all other deposits to a very insignificant position. Should a new use for colemanite be developed, other than as source material for refined borax, the deposits in California and Nevada could still supply a large demand.

The production of borate minerals in the United States has thus had four successive distinct periods, varying in location of source material and mineral composition of material mined. These may be summarized as follows:

First period	Northern California, 1864-1872	Borax
Second period (Marsh period)	Western Nevada and Southern California, 1872-1887	Borax and cotton-ball ulexite
Third period (Colemanite period)	Southern California and Southern Nevada, 1887-1926	Colemanite and massive ulexite
Fourth period	Southern California 1926 (Kramer and Searles Lake)	Borax, kernite, and brine

#### PRODUCTION AND CONSUMPTION

From first production in about 1864 to 1883 (discovery of colemanite), the annual production was less than 3000 short tons of refined borax. For the next 15 years it varied between 3250 and 8000 tons per annum. Beginning with 1899, the annual production of crude borates increased considerably through 1913, fluctuating between 18,000 and about 60,000 short tons. Beginning with 1914, more than 60,000 short tons was produced annually, except for 1921 (50,000 tons). By 1923 both world and domestic production had reached a high figure, world production in that year being slightly over 200,000 short tons, a figure not again reached until 1933. Between 1923 and 1934 the annual domestic production ranged from 100,000 to 200,000 short tons, but in 1934 and 1935 production exceeded 200,000 short tons.

There has thus been a steady increase in the production of borates in the United States, with the present quantity far exceeding that of any previous time. No other country has shown such a steadily increasing output. The development of the two producing localities in California, both furnishing sodium borates, has adversely affected all other important deposits of borates throughout the world. Only the boric acid production in Italy has retained or improved its position.

In consequence, the percentage of domestic production to world production has increased from an average of 65 per cent for the years 1920 to 1928, inclusive, to an average of 93 per cent for the years 1929 to 1935. For both 1934 and 1935 the United States produced 95 per cent of all borates mined throughout the world.

The domestic and world production of crude borates for the years 1920 to 1935, inclusive, is shown graphically in Fig. 1.

For a long time Chile was the second largest producer of crude borates (ulexite), furnishing 55,000 short tons in 1913 and exceeding the production of the United States in 1908 and 1912, and nearly equaling it in other years. Argentina, Bolivia and Peru have produced much smaller quantities of ulexite (a few thousand tons annually). Production of crude borates from Chile declined rapidly after 1926 and production in South America practically ceased in 1929.

Turkey has been the third largest producer, furnishing about 10,000 short tons of priceite annually. Production declined about 1930,

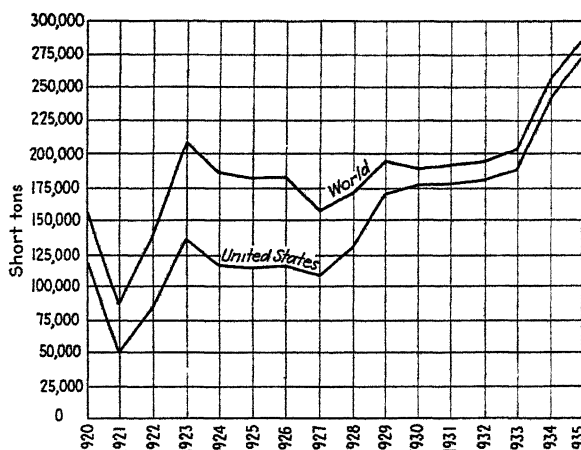


FIG. 1.—WORLD AND DOMESTIC PRODUCTION OF CRUDE BORATES, 1920-1935.

but, including 1934, amounted to 6000 to 7000 short tons annually. Italy has not only maintained its yield of 2000 to 4000 short tons of boric acid annually but since 1930 has increased it to between 5000 and 6000 tons. A total of about 1000 tons of borax from Tibet and boracite from Germany has been produced annually.

Around 1920 the apparent domestic consumption was about equal to domestic production. Beginning with 1923, exports of borax increased considerably, with a still further increase from 1927 on. From 1920 to 1926, inclusive, the United States consumed over 90 per cent of the domestic production. With the development of the present two producing localities near Kramer, and at Searles Lake, domestic production increased rapidly from 1928 on and exports likewise increased, so that from 1928 to 1935, inclusive, the apparent domestic consumption dropped to between 50 and 60 per cent of domestic production. This means that the two producing localities in California are supplying not only domestic requirements but nearly the entire world's demand for borax and related boron compounds.

## MINING METHODS AND PREPARATION FOR MARKET

Each of the three deposits of minable borates near Kramer consists of about one-quarter clay and three-quarters borate of soda. In the two deposits containing kernite, that mineral and borax are present in nearly equal proportions. Each of the deposits occurs as nearly horizontal tabular bodies or beds of borates parallel to the bedding of the enclosing clays and shales. After the shafts have penetrated the overburden the drifts and tunnels are cut in the borate bodies. In the one operating mine (1934) a regular set of drifts leads off from the main tunnels on the several levels and everything mined is borate. Considerable pillars are left for support but these are as rich as the material removed.

A selection either of nearly pure borax or of kernite (except for the ever present clay) could be mined, but it was found better to mine and grind the two minerals together. When crushed, kernite breaks into fine fibers, as the mineral possesses three cleavages in one zone. The granular borax prevents these cleavage fibers from clogging the grinders. The mixed borates are ground and roasted to remove water (26 per cent in kernite and 47 per cent in borax) and most of the clay is separated before shipment to Wilmington, Calif., for further refining.

On the basis of mineral composition as given above, the material mined should furnish about 90 per cent of refined borax by weight, without allowance for refining losses. The high yield is due to the fact that the change of kernite (with  $4\text{H}_2\text{O}$ ) to borax (with  $10\text{H}_2\text{O}$ ) consists in the addition of water, so that pure kernite, free from clay and other impurities, would yield theoretically 1.39 times as much borax. In actual mining procedure, it is not possible to obtain large quantities of kernite free from clay, and the advantage of having borax present for the grinding has already been mentioned.

After the discovery of borax at Searles Lake, a small plant operated intermittently until about 1895. In 1912 the production of potash salts and borax was seriously considered, and during the war years potash salts were extracted. Later, following an extensive program of research, the commercial production of borax from the brines was started in 1919, and in 1920 a total of 4643 tons of borax was produced, increased by 1926 to 17,500 tons and by 1930 to more than 50,000 tons.

At Searles Lake, the unusual chemical composition of the brine presented many operating problems and the physical chemistry of the equilibrium relations existing between the various salts in solution had to be worked out on the basis of temperature and concentration relationships. An extensive program of research was undertaken to obtain the fundamental knowledge of the relationships of these various salts,

necessitating changes in the previous manufacturing process and in equipment.

The process used for the production of borax, potassium chloride and other salts is essentially one of evaporation followed by fractional crystallization. The temperature and concentration of the brine must be accurately controlled throughout each step and the results obtained are an outstanding example of the usefulness and accuracy of the application of the results of physical-chemical research to industrial manufacturing problems.

After the brine is removed by wells sunk in the porous salt layer, it is mixed with the various end solutions from previous operations, and fed into evaporators or vacuum pans. The larger portion of the carbonate, sulphate, and chloride of sodium is precipitated during evaporation and removed. When the brine is nearly saturated with potassium chloride, it is rapidly cooled and the potassium chloride separates out. On further cooling, borax and other salts crystallize out, which by resolution and controlled cooling and crystallization yield pure borax.

#### USES AND PRICES

Borax is widely used for many purposes. Its easy solubility in water to a mildly alkaline and antiseptic solution, its low melting point and excellent fluxing properties make it one of the most useful of salts. Best known, perhaps, as a household commodity, it is of more importance in manufacturing industry, and many articles of everyday life require borax in their manufacture.

An excellent cleansing agent, it is widely used in industrial cleansing, as in washing, cleansing and laundering, either directly or as a constituent of soaps and soap powders.

Both borax and boric acid find many applications in medicine and pharmacy, as in disinfectants, mouth washes, tooth powders, cosmetics, lotions, ointments, deodorants and medicated lint and gauze.

Water solutions of borax are used to dissolve casein, which is extensively used in the manufacture of coated papers, playing cards, plywoods, plaster, paints and calcimines. The mild antiseptic character of borax prevents rancidity in cosmetics, pastes, glues, and various materials, and prevents the formation of mold or mildew in starches and sizes, of citrus fruits, leathers, textiles, etc. It is employed to retard the growth of certain fungi in lumber, and for fireproofing wood, paper and fabrics, for degumming silk, for neutralizing free acid in leather, and in dyeing leather and textiles. It hastens the cleansing of hides and skins and boric acid produces a soft, smooth grain in finished leather.

Its uses extend to baking powder, food preservatives, flavoring extracts, sirup and pickle manufacture, insect repellents, and many



others. Almost the whole line of manufacturing products finds use for borax, as in the manufacture of various kinds of implements, candles, carpets, drug specialties, dyes, hats, ink, jewelry, oil, paints, paper, stove polish, shoes, tobacco, tools, and many others.

Borax is an essential constituent of porcelain enamels, coating iron and steel as applied to kitchen utensils, stoves, refrigerators, washing machines, bathtubs, table tops, sinks, signs, pipe lines, tiles, and an endless array of products requiring an attractive, durable, and sanitary finish. The borax also facilitates the addition of coloring pigments to the enamels. In the manufacture of pottery, bricks, china, and both white and colored ceramic ware, glazes, and many kinds of glass, borax is indispensable, as it lowers the coefficient of expansion and makes the product more durable. Thus heat-resisting glasses are used in the kitchen and laboratory, for lamp chimneys, signal lenses, thermometers and optical glasses. In recent years the glass industry has become one of the largest consumers of borax, as it aids in the melting process and increases the strength, brilliance and durability of glass.

It is a good flux in the brazing, welding, soldering, etc., of metals, in assaying, in smelting copper and in refining gold and silver. Certain borides are good deoxidizers for nonferrous metal melts, such as brasses and bronzes. Ferroboration, in small quantities, is a deoxidizer for steel. Boron steels, with less than 2 per cent boron, are claimed to possess remarkable strength.

Boric acid, also water-soluble and antiseptic, is used in eyewashes, for hair waving, and other ways.

Borax glass, calcined borax, and anhydrous boric acid are also useful. Manganese borate is used as a drier in the manufacture of drying oils, linoleum, varnishes, printing and stamping inks. Ammonium borate is used in hair waving and certain types of electrolytic rectifiers and condensers. Boron carbide is extremely hard and is used as an abrasive. Sodium perborate is a useful disinfectant and solutions of it are used in dyeing and bleaching, and in electroplating.

Thus the cleanser, the pharmacist, the paper and textile manufacturer, the tanner, the potter, the enameler, the glassmaker and tilemaker, the metallurgist, the brazier, the jeweler, all use borax. Hardly any other substance enters into so many diversified lines of manufacture.

The value of the first refined borax produced from northern California ranged between \$700 and \$800 per short ton, or about 36¢ per pound. After the borax marshes of western Nevada and southern California began to produce, and the large deposits of colemanite were worked, the values of refined borax dropped rapidly, reaching a then low value of \$100 per short ton (or 5¢ a pound) in 1886, 1887, 1895 and 1896. No statistics on the value of refined borax are available for a number of

years but from 1914 to 1923 it varied from about \$80 to \$160 (in 1920) per ton. From 1920 on the value dropped, to \$30 a ton in 1931 and about \$35 a ton in 1935. From 1903 on, production and value of crude borate minerals is given, and this has ranged generally between \$20 and \$40 a short ton, with an average value of \$26 for the period 1920-1935. Since 1927 the value has not exceeded \$30 and for the last four years has been close to \$20 a ton.

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## CHAPTER IX

### CEMENT MATERIALS

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SO extensive has become the use of cement in highway and building construction in recent years that it has been suggested that the present stage of civilization be designated the "cement age." In this development and expansion of the cement industry quantitatively, qualitatively and geographically, numerous raw materials have been utilized. Some of these, used in the manufacture of highly specialized varieties of cement, find their chief application in other fields and are described elsewhere in this volume. This article therefore deals only with the raw materials of the puzzolan, natural and Portland cements.

#### PUZZOLAN CEMENT

Puzzolan cement is so named from Pozzuoli, Italy, where a volcanic ash, called *pozzuolana*, has been dug, pulverized and mixed with quicklime to form a cement. This is one of the oldest cements known and was extensively used for roads and other structures at a very early date in Roman history. Similar volcanic rocks from other parts of the world have been used, especially in Europe. This type of cement possesses hydraulic properties by which it hardens or sets under water. The action of the quicklime on the volcanic products containing aluminum silicates produces lime silicates not unlike those of Portland cement. At a later date slags from the blast furnace were substituted for the volcanic ash, with satisfactory results.

The analysis of *pozzuolana* (volcanic ash) is:  $\text{SiO}_2$ , 44.50 per cent;  $\text{Al}_2\text{O}_3$ , 15.00;  $\text{Fe}_2\text{O}_3$ , 12.00;  $\text{CaCO}_3$ , 8.80;  $\text{MgCO}_3$ , 4.70; alkalies, 5.50; loss on ignition, 9.50. Puzzolan cements are variable in composition and in quality. Although some Roman objects of this material have survived hundreds of years, they are distinctly inferior to the modern Portland cement and are much more variable in chemical composition and in physical characteristics. A few years ago the writer had opportunity to examine some of the Roman cement baths discovered in the process of uncovering the ancient city of Pompeii. The cement showed no signs of deterioration and was actually harder than most of the natural stones of that region, although 2000 years old. For certain uses, puzzolan cements

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are perfectly satisfactory and are still used. Where they can be produced at low cost they will continue to be manufactured. In the United States their production has never assumed much importance.

### NATURAL CEMENTS

Natural cements are of more recent origin than puzzolan but antedate Portland by 68 years. Natural cement, as indicated by the name, is a product made by burning a clayey limestone in its natural state. John Smeaton, an English civil engineer, in 1756 found that "lime" made by burning an argillaceous limestone possessed the property of hardening under water. He stated that lime made from limestones containing from  $\frac{3}{14}$  to  $\frac{1}{17}$  of clay in "chemical combination" possessed these hydraulic properties. He said "it remains a curious question which I must leave to the learned naturalist and chemist, why an intermediate mixture of clay in the composition of limestone of any kind, either hard or soft, should render it capable of setting in water in a manner no pure lime I have yet seen, from any kind of stone whatsoever, has been capable of doing" (ref. 3, p. 3).\*

Smeaton's work led to many investigations and during the next 60 years a great many varieties of natural cements were manufactured in

TABLE 1.—*Analyses of Magnesian Limestones from Rosendale, N. Y., Used in Manufacture of Natural Cement*<sup>14</sup>

	PER CENT				
	1	2	3	4	5
SiO <sub>2</sub> . . .	30.50	29.98	30.84	27.30	27.98
Al <sub>2</sub> O <sub>3</sub> . . .	6.84	6.88	7.75	7.14	7.28
Fe <sub>2</sub> O <sub>3</sub> . . .	2.42	2.50	2.11	1.80	1.70
CaCO <sub>3</sub> . . .	34.38	33.23	34.49	35.98	37.59
MgCO <sub>3</sub> . . .	18.00	17.80	17.77	18.00	15.00
Alkalies . . . . .	3.98	7.10	4.00	6.80	7.96
Loss on ignition . .	3.78	3.13	3.04	2.98	2.40

England and France, and various patents were issued. Instead of a single type of stone, a number of combinations were employed. Usually clay or shale was mixed with limestone, chalk or marl. In most cases the burning was carried little beyond the temperature necessary for the elimination of the CO<sub>2</sub>, although some manufacturers increased the temperature almost to the point of fusion.

The earliest production of natural cements in the United States is associated with the construction of locks for canals in the early part of the nineteenth century. When the Erie Canal was dug in 1818-1819 a

\* References are at the end of the chapter.

successful search was made along the route for stone satisfactory for the manufacture of natural hydraulic cement, and a few years later, when the Lehigh Coal and Navigation Canal was built along the Lehigh and Delaware Rivers in Pennsylvania, similar rock was sought and cement plants were erected at several places. At Rosendale, N. Y., so much natural cement has been produced that the name "Rosendale cement" has become almost a synonym for "natural cement" and has been used in some instances to designate natural cements made in other places. Analyses of Rosendale limestones are given in Table 1.

Natural cements were made extensively in the Lehigh Valley and elsewhere long before Portland cement. Production continued after the establishment of the Portland cement industry, although steadily declining until at present it is of relatively little importance. Owing to the lack of control of the raw mixture as well as the degree of burning, the resulting product is variable and can find a market only when it can be sold at a substantially lower cost than Portland cement. In Table 7, it may be noted that the average price of natural cement in the United States in certain years is higher than that of Portland cement. This is explained by its proximity to the source of consumption and the greater distance to Portland cement plants. Transportation costs constitute a large part of the selling price of cement. This situation at present exists in comparatively few places. Nevertheless there are some who maintain that for certain specific purposes some of the higher grade natural cements possess distinct advantages over Portland cement.

Rock not suitable for the manufacture of Portland cement can be used in natural cements. In general, there is a higher percentage of silica and magnesia. Some stone that has been used is practically pure dolomite and in many cases the  $\text{MgCO}_3$  content runs from 20 to 35 per cent, whereas stone containing less than 10 per cent is demanded for the manufacture of Portland cement, and the lower the magnesia the more desirable it is. The silica likewise varies greatly in the rocks used for natural cements. In many it exceeds 20 per cent and occasionally rock with more than 30 per cent has been used.

It is evident that natural cements can be made over a wider area than Portland cement, since all argillaceous limestones suitable for the Portland variety are equally adaptable for natural cement as well as other rocks that are useless for Portland cement. For this reason the manufacture of natural cements still continues in some sections.

#### PORTLAND CEMENT

Portland cement now amounts to over 99 per cent of the total annual cement production.

Joseph Aspdin, a bricklayer of Leeds, in the County of York, England, is credited with having been the discoverer of Portland cement. On

Dec. 15, 1824, he applied for a patent for a product which he described as follows:

My method of making a cement or artificial stone for stuccoing buildings, waterworks, cisterns, or any other purpose to which it may be applicable (and which I call Portland cement) is as follows: I take a specific quantity of limestone, such as that generally used for making or repairing roads, and I take it from the roads after it is reduced to a puddle, or powder; but if I cannot procure a sufficient quantity of the above from the roads, I obtain the limestone itself, and I cause the puddle or powder, or the limestone, as the case may be, to be calcined. I then take a specific quantity of argillaceous earth or clay, and mix them with water to a state approaching impalpability, either by manual labour or machinery. After this proceeding I put the above mixture into a slip pan for evaporation, either by the heat of the sun or by submitting it to the action of fire or steam conveyed in flues or pipes under or near the pan until the water is entirely evaporated. Then I break the said mixture into suitable lumps, and calcine them in a furnace similar to a lime kiln till the carbonic acid is entirely expelled. The mixture so calcined is to be ground, beat, or rolled to a fine powder, and is then in a fit state for making cement or artificial stone. This powder is to be mixed with a sufficient quantity of water to bring it into consistency of mortar, and thus applied to the purposes wanted.<sup>2</sup>

The product that Aspdin made was apparently a poor cement when compared with the Portland cement of today. He did not seem to recognize the importance of burning the mix to incipient fusion nor did he explain the meaning of "specific quantity of argillaceous earth, or clay," which indicates that the proportions of the ingredients were only roughly determined. Nevertheless, his Portland cement was an improvement on the other types of cement previously used. The name Portland was chosen either because the cement resembled Portland stone or it was intended as a substitute for that widely used building stone.

During the next 30 to 40 years, manufacture of Portland cement extended to other European countries; first to Belgium and then to France, Germany and Austria. Imports of Portland cement came into the United States in increasing amounts during the Civil War and later, and entered into competition with natural cements produced in several localities.

As the result of rather crude experimentation, David O. Saylor, a manufacturer of natural cement in the Lehigh Valley, Pennsylvania, in 1871 succeeded in making a product similar to the imported Portland cement. On Sept. 26, 1871, he received a patent on a process and product which he described as follows:

I have discovered that some kinds of the argillo-magnesian and also argillo-calcareous limestone found along the Appalachian range, containing more or less carbonate of lime, magnesia, silica, alumina, iron, salts, and alkalies adapted to the purpose, and which are now extensively used in the manufacturing of hydraulic cement, will make, when burned to a state of incipient vitrification, so as to be agglutinated, warped, or cracked, by contraction, and some burned to cinders, a very superior and heavy hydraulic cement, weighing from one hundred and ten pounds to one hundred and twenty pounds per bushel, and in every respect equal to the Portland cement made in England and imported into this country.<sup>11</sup>

Saylor's Portland cement was not always uniform and satisfactory but in 1876 at the Centennial Exposition in Philadelphia it received an "award of merit." Since that time the Portland cement industry of the United States has steadily progressed. The production has increased and the quality has been improved.

There have been a number of definitions proposed for the modern Portland cement, and from time to time the accepted definitions or specifications in different countries have been substantially modified. The definition accepted by the American Society for Testing Materials in 1933 is as follows:

Portland cement is the product obtained by finely pulverizing clinker produced by calcining to incipient fusion an intimate and properly proportioned mixture of argillaceous and calcareous materials, with no additions subsequent to calcination except water and calcined or uncalcined gypsum.

In addition there are a number of chemical and physical requirements.

#### CONSTITUTION OF PORTLAND CEMENT

A large amount of chemical and petrographic work has been done on Portland cement but there still remain many questions in regard to its chemical and mineralogical composition. It has been proved that it is a mixture of several compounds. The essential ingredients are  $\text{CaO}$ ,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ . These combine in the kiln to form silicates and aluminates, which at varying temperatures break down and recombine to form still other compounds. Rankin and Wright<sup>13</sup> have shown that a variety of products can be formed from these oxides alone or by varying combinations of two or all three. They list 26 different combinations, any one of which may develop, and give a table of optical and crystallographic properties. Some are stable only at certain temperatures whereas others are stable at all temperatures. At specific temperatures particular combinations result.

Only a relatively small number of these possible compounds have been identified in Portland cement. When the correct proportions of  $\text{CaO}$ ,  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  are used and the mixture is properly burned, the clinker should be composed of tricalcium silicate ( $3\text{CaO} \cdot \text{SiO}_2$ ), beta form of dicalcium silicate ( $2\text{CaO} \cdot \text{SiO}_2$ ), and tricalcium aluminate ( $3\text{CaO} \cdot \text{Al}_2\text{O}_3$ ). A number of the other combinations of the three oxides of Ca, Si and Al have been noted in finished Portland cement as well as the uncombined oxides.

Inasmuch as the rocks used in the Portland cement industries generally contain some iron, magnesia, alkalies, as well as other materials, naturally there are other compounds produced, some of which have received rather careful study.  $\text{MgO}$  does not readily unite with either  $\text{Al}_2\text{O}_3$  or  $\text{SiO}_2$  at the normal kiln temperature, so that it is regarded as an undesirable ingredient and consequently any appreciable amounts present in the raw



materials unfit the stone for use. A number of investigators claim that the MgO passes through the kiln and into the clinker entirely uncombined. This is disputed by others, who report the MgO entering into combination in similar manner to CaO and forming the compounds  $2\text{MgO} \cdot \text{SiO}_2$ ,  $2\text{CaO} \cdot \text{MgO} \cdot 2\text{SiO}_2$ ,  $3\text{CaO} \cdot \text{MgO} \cdot 2\text{SiO}_2$ , and  $\text{MgO}(\text{Al}, \text{Fe})_2\text{O}_3$ . The permissible limit of 5 per cent MgO has been fixed for the finished cement. In making a mix,  $\text{Fe}_2\text{O}_3$  can be substituted for  $\text{Al}_2\text{O}_3$ . For certain kinds of Portland cement rather large percentages of iron oxides are added to the mix instead of clay or shale. Normally there is some iron in almost all limestones, shales and clays. There are several iron compounds in which  $\text{Fe}_2\text{O}_3$  unites with CaO or with both CaO and  $\text{Al}_2\text{O}_3$ . The most common compound is tetracalcium aluminoferrite ( $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ ).

The alkalies enter into chemical combinations to a slight degree. They are regarded as undesirable. Most of the potash appears to be volatilized and passes off through the stack. During the World War some cement plants installed apparatus to collect the flue dust for fertilizing purposes, because of its high content of potash.

Altogether the constitution of the various grades and types of Portland cement is still a fruitful field of research. A great deal of research work has been done in the chemical reactions that take place in the hardening or setting of cement and on the minerals developed. Most of the conclusions are highly theoretical and many are definitely in conflict. Kühl reaches the following conclusion as to the hardening process that takes place when water is added to the finished Portland cement:

It has thus been convincingly proved that, if we ignore the unimportant minor constituents, hardened cement mortar must be regarded as an unstable gel consisting of dicalcium hydrosilicate, tricalcium hydroaluminat, hydrated lime and calcium ferrites of unknown composition. All these ingredients tend slowly to pass over into the crystalline form, although this transformation requires a period of some years before it can be observed and some decades before it is complete. We are ignorant of the order in which the different gel compounds assume a microcrystalline structure. Probably the calcium hydroxide and calcium hydroaluminat are first affected, followed only at a later stage by the gradual transformation of calcium hydrosilicate.<sup>6</sup>

*Desirable Proportions of Ingredients for the Kiln Mix.*—It is not feasible to enter into a full discussion of the composition of the mix as it is fed to the kiln, inasmuch as there are so many varieties of Portland cement now being manufactured, each of which may have definite specifications. Since it is believed that the tricalcium silicate ( $3\text{CaO} \cdot \text{SiO}_2$ ), which contains by weight 73.6 per cent CaO and 26.4 per cent of  $\text{SiO}_2$ , is the most important constituent of Portland cement, obviously rocks that can furnish these materials in somewhat this percentage might be regarded as most desirable. However, in practice this does not hold, as siliceous limestones cannot be used alone for the manufacture of Portland cement.

As a generalized statement, it may be said that the raw mix should contain about 75 per cent  $\text{CaCO}_3$ , about 20 per cent  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ ,

with about 5 per cent for the impurities such as magnesia, alkalis, etc. The silica should approximately equal the combined  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$ .

### ROCKS UTILIZED IN MANUFACTURE OF CEMENT

Since Portland cement is a cheap product, until recently it has not been economically feasible to break up different kinds of rocks, selecting the desirable materials and discarding the undesirable. Therefore the cement manufacturers have sought some natural rocks in which nature has combined the ingredients in the proper proportions for the best grade of cement. Failing in this, they have looked for a second kind of rock of such composition as to make up the deficiencies. Joseph Aspdin first used a mixture of Carboniferous limestone and alluvial clay and later

TABLE 2.—*Materials Used in the Cement Industry*

Source of $\text{CaO}$	Source of $\text{Al}_2\text{O}_3$	Source of $\text{SiO}_2$	Source of Fe
Limestones.	Clay	Clay	Limestones and marls
Calcareous marls	Shale	Shale	Clay, shale
Fossil-shell marls (bog lime)	Slate	Slate	Slate
Recent shells	Ash from coal used as fuel	Ash from coal used as fuel	Argillaceous limestones
Alkali waste	Argillaceous limestones	Argillaceous limestones	Iron ores
Blast-furnace slag	Blast-furnace slag	Blast-furnace slag	Blast-furnace slag
	Granite	Sand	
	Andesite	Sandstones	
		Granite	
		Andesite	

chalk and clay. The first Portland cement of the United States was by pure luck made of an argillaceous limestone that is well developed throughout the Lehigh Valley, and which has in many places almost exactly the requisite proportions of the necessary constituent minerals. Few regions are as well favored. Sometimes one finds a property where strata of different compositions can be quarried to make a proper mix. Other companies are required to obtain part of the necessary stone from fairly distant places. Since the finished product is cheap in price and the margin of profit small, it is not economically possible to pay freight charges on any considerable part of the stone.

When it becomes necessary to bring together two or more kinds of rock for the proper mix, the prevailing custom is to use a calcareous rock to supply the necessary calcium oxide and an argillaceous material for the alumina and silica. Sometimes a siliceous rock such as sand or sandstone

is used, bauxite when more alumina is desired and iron ores if a high-iron cement is produced. The list of materials given in Table 2 may not be absolutely complete, but it is believed to include all the sources that have been utilized to a large extent.

#### LIMESTONES\*

A limestone may be defined as a sedimentary rock, normally white to light gray or buff in color, composed primarily of calcium carbonate ( $\text{CaCO}_3$ ) and occurring in beds or strata.

*Chemical Composition.*—From the normal type, there are many variations and in places limestones pass almost insensibly by gradation into shales or sandstones. No one has attempted arbitrarily to draw the lines separating the various classes of sediments. Instead, it is customary to use adjectives to describe the intermediate varieties. Thus we have siliceous limestones or calcareous sandstones and argillaceous limestones or calcareous shales. In addition, limestones containing abnormally large percentages of iron, phosphate, carbonaceous matter and magnesia are described respectively as ferruginous, phosphatic, carbonaceous and magnesian limestones. In some cases, the presence of ingredients other than calcium carbonate tends to make the limestones less valuable, but in others foreign matter renders them more useful for particular purposes.

A pure limestone should contain 100 per cent of  $\text{CaCO}_3$ , and there are a few that approximate this composition. Since virtually all our limestones have been laid down in shallow ocean waters in which there are various substances other than  $\text{CaCO}_3$  in solution and in suspension, it is to be expected that some of these will be precipitated on the ocean bottom.

In analyzing limestone, the substances usually determined are calcium oxide ( $\text{CaO}$ ) or calcium carbonate ( $\text{CaCO}_3$ ), magnesium oxide ( $\text{MgO}$ ) or magnesium carbonate ( $\text{MgCO}_3$ ), carbon dioxide ( $\text{CO}_2$ ), silica ( $\text{SiO}_2$ ), aluminum oxide ( $\text{Al}_2\text{O}_3$ ), ferric oxide ( $\text{Fe}_2\text{O}_3$ ), ferrous oxide ( $\text{FeO}$ ), sulphur ( $\text{S}$ ), sulphur dioxide ( $\text{SO}_2$ ), sulphur trioxide ( $\text{SO}_3$ ), phosphorus ( $\text{P}$ ) or phosphorus pentoxide ( $\text{P}_2\text{O}_5$ ), calcium sulphide ( $\text{CaS}$ ), strontium oxide ( $\text{SrO}$ ), hydrogen sulphide ( $\text{H}_2\text{S}$ ) and water ( $\text{H}_2\text{O}$ ). For individual purposes the analysis will not show nearly all of these. For example, a steel company may determine only the amount of silica and a cement company rarely determines other than the calcium, magnesium, silicon, iron and aluminum compounds. The substances occasionally determined are manganese dioxide ( $\text{MnO}_2$ ), titanium oxide ( $\text{TiO}_2$ ), carbon ( $\text{C}$ ), sodium oxide ( $\text{Na}_2\text{O}$ ), and potassium oxide ( $\text{K}_2\text{O}$ ).

It is recognized that all of these materials may and do vary widely in the amount present. It is not possible to state the limits definitely

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\* A portion of the general descriptions of limestone is taken from the author's *Limestones of Pennsylvania*, Bull. M20, Pennsylvania Geol. Survey 4th ser. (1934). 729 pp.

but in general the following ranges are believed to be approximately correct. The  $\text{CaO}$  is apt to vary from 22 to approximately 56 per cent in some of the purest limestones. These correspond to 39.29 and 100 per cent of  $\text{CaCO}_3$ . The  $\text{MgO}$  content may vary from 0 to 21.43 per cent. This corresponds to 0 to 45 per cent of  $\text{MgCO}_3$ . In a few instances the magnesium has been reported as even higher, which probably means that some is present uncombined with calcium carbonate. Some analyses of this kind known to the author, however, are regarded as of questionable accuracy.

The  $\text{Al}_2\text{O}_3$  varies from zero in the pure limestones to as much as 5 or 6 per cent, or occasionally more, in the argillaceous limestones used for the manufacture of Portland cement. By a still further increase of  $\text{Al}_2\text{O}_3$  and a corresponding decrease in  $\text{CaCO}_3$ , the rock becomes calcareous shale and is no longer appropriately called a limestone.

The  $\text{Fe}_2\text{O}_3$  and  $\text{FeO}$  range up to several per cent in some of the ferruginous limestones. Seldom do the iron oxides exceed 3 or 4 per cent. However, in western Pennsylvania the Vanport limestone has been changed to siderite by replacement and all gradations can be found from high-grade limestone ( $\text{CaCO}_3$ ) to siderite ( $\text{FeCO}_3$ ). When the siderite undergoes oxidation, as it does readily, the resulting material is practically pure iron oxide. Locally limestones are found unusually high in pyrite. On analysis, the iron is apt to be reported as oxide and may be higher than given above. Any limestone with more than 1 or 2 per cent of iron oxide is apt to become brown or red on oxidation by exposure to the air.

The silica content in limestone is very important for many industries using limestone. It is determined in almost every limestone analysis and may be said to be present almost universally. In many of the purest limestones it constitutes much less than 1 per cent. It probably averages from 3 to 6 per cent, usually more in the dolomitic limestones than in the low-magnesium varieties. In the most siliceous limestones, silica constitutes about 50 per cent of the stone. In certain places the Loyalhanna limestone of Mississippian age passes by gradation into a true sandstone in which the silica constitutes practically the entire composition.

Sulphur is frequently noted in chemical analyses of limestones and occasionally it is reported as  $\text{SO}_2$  or  $\text{SO}_3$ . It is unusual to find more than 1 or 2 per cent of sulphur present and generally it is much less than 1 per cent. The sulphur present, although generally reported as uncombined, is almost altogether in combination as sulphides, principally pyrite ( $\text{FeS}_2$ ) or marcasite ( $\text{FeS}_2$ ), or as sulphates such as gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) or strontium sulphate ( $\text{SrSO}_4$ ).

Phosphorus, reported as P or as  $\text{P}_2\text{O}_5$ , is generally negligible in limestones although commonly present in extremely small amounts. It is generally regarded as objectionable but the permissible amounts have not been satisfactorily determined. Its effect in fluxing stone is known,

but not in Portland cement and other manufactured products. Some unaltered limestones in Kentucky and Tennessee contain from 2 to 6 per cent of  $P_2O_5$ , according to report. One cement company is said to have had difficulty in producing a satisfactory grade of Portland cement, because of the large amount of phosphorus in the stone used.

The water content of limestones is extremely variable. In most cases the analyses are reported as water-free. The amount of carbon present also varies greatly. It may occur in sufficient amount to make the limestone black, yet because of the ease with which it is oxidized and eliminated in manufacturing plants, it is seldom determined even though it may constitute several per cent of the stone. The other constituents of limestones are almost invariably present in such small amounts, fractions of one per cent, that there is little occasion to regard them, and their presence does not affect the utilization of the stone.

*Mineralogical Composition.*—The chemical composition of a stone does not necessarily indicate the particular minerals present. The minerals are definite compounds and some of the chemical elements and combinations as reported in chemical analyses may enter into several different minerals. For example, the silica may, and usually does, exist mainly as quartz, which is pure silica, but in addition part of it is combined into various silicate minerals; sulphur may be present uncombined, but is almost always united with iron to form pyrite or marcasite, with some other metals to form other sulphides, or with metals and oxygen to form sulphate minerals. In a similar way, almost every substance determined by the chemist may enter into combination with other material to form a number of different minerals.

The desirability of a particular stone may depend upon the minerals present and often may not be determined by the chemical analysis. An illustration is furnished by pyrite and marcasite, both with the same composition ( $FeS_2$ ) and yet with many different properties. On exposure to moist air both will oxidize to form iron oxides and sulphuric acid but with different rapidity. Marcasite will decompose quickly and a stone containing a large amount may crumble in a short time because of this action, whereas pyrite undergoes such a change very slowly in comparison. Other illustrations might be presented to show the importance of knowing the mineralogical composition of a stone as well as its chemical constitution.

A great variety of minerals has been reported as occurring in limestones. The list in Table 3 is not claimed to be complete but doubtless it includes most of the minerals thus far recognized. Of these calcite is the only essential mineral of pure limestone, and calcite and dolomite the only essential ones forming dolomitic limestones.

*Methods for Distinguishing Calcite and Dolomite.*—It is frequently useful to determine whether a limestone is composed mainly of calcite or dolomite and if possible to do so in the field. As the chemical analysis

of limestone is expensive and requires time, attempts have been made to locate stone of the required kind with a minimum of chemical analyses. Considerable progress has been made, although most geologists feel that they must check their field determinations by occasional analyses. Several methods are in common use.

If dolomite is present in large amounts, frequently the amount by weight can be approximated by balancing in the hand, as dolomite is slightly heavier than calcite. Of course, this method requires experience and at best is a rough determination. The color is apt to be suggestive in materials low in iron and carbonaceous matter. The more highly magnesian limestones show a light bluff tinge whereas the low-magnesia varieties are white or bluish gray. On weathering, however, the dolomitic layers become chalk white, considerably whiter than the low-magnesia strata.

The different degrees of solubility are more reliable in that calcite dissolves fairly readily in cold dilute hydrochloric or acetic acid and produces vigorous effervescence, while dolomite dissolves and effervesces very slowly in comparison. However, if the dolomite is pulverized by the blow of the hammer or if the rock is feebly cemented, and hence porous, this test is not serviceable because the effervescence may be almost as rapid and vigorous as in the low-magnesia rocks.

The low-magnesia limestones are soft and easily broken in comparison with the highly dolomitic ones, so that a geologist can with practice rather closely approximate the magnesian content by the hardness and toughness of the stone when struck with the hammer. The dolomitic stones are also finer grained and more compact than those lower in magnesia of the same region.

When weathered surfaces are available the distinctions between high-magnesia and low-magnesia limestones can readily be made. The high-magnesia limestones contain numerous straight cracks running in all directions, along which vein material has commonly been deposited in layers so extremely thin that the freshly broken surface scarcely indicates their existence. On being exposed to weathering agents, these cracks furnish access to dissolving fluids, and the weathered surface of the rock looks as though someone had hacked the stone with a steel-cutting implement. Where high-magnesia and low-magnesia limestones are interbedded, the contrast on weathered surfaces is striking. There is also a greater tendency for dolomitic limestones to show a distinctly granular surface on weathering.

Where interbedded high-magnesia and low-magnesia limestones have been deformed by folding, the dolomitic limestones have many more gash veins of quartz and calcite than do the purer limestones. There is also commonly a greater proportion of quartz in the vein fillings of the dolomites in comparison with calcite and dolomite.

Where weathering has progressed downward to considerable depth, the dolomites in various places of great crustal disturbances appear to be shattered as though by a blast of dynamite. One quarry, near Lancaster, Pa., in a highly dolomitic limestone region, was worked for road metal by means of a steam shovel without any blasting.

The highest grade of limestones, composed almost entirely of calcium carbonate, can be found in certain regions by noting the sinkholes and caves, caused by their ready solubility or by heavy clay overburden. In such instances, outcrops are apt to be few.

All of these shortcuts to the determination of the chemical composition of limestones by means of physical characteristics are useful, but must be used with care, as the most expert geologists in this line occasionally go astray, especially on entering a new region.

Since froth flotation has been used in the cement industry it has become important to know of the limestone minerals that have been reported. The list in Table 3 is fairly complete and includes a number that at present seem to have little economic importance.

TABLE 3.—*Minerals of Unmetamorphosed Limestones*

Carbonates	Silicates
Calcite ( $\text{CaCO}_3$ )	Feldspars
Aragonite ( $\text{CaCO}_3$ )	Albite ( $\text{NaAlSi}_3\text{O}_8$ )
Dolomite ( $\text{CaCO}_3 \cdot \text{MgCO}_3$ )	Microcline ( $\text{KAlSi}_3\text{O}_8$ )
Siderite ( $\text{FeCO}_3$ )	Micas, especially muscovite or sericite
Ankerite [ $\text{CaCO}_3 \cdot (\text{Mg, Fe, Mn})\text{CO}_3$ ]	[ $\text{H}_2\text{KAl}_3(\text{SiO}_4)_3$ ]
Oxides	Tourmaline (complex silicate of boron and aluminum with magnesium, iron or alkali metals)
Quartz ( $\text{SiO}_2$ )	Garnet (andradite type) [ $\text{Ca}_3\text{Fe}_2(\text{SiO}_4)_3$ ]
Chert, flint, basanite, chalcedony, etc. (all are composed of $\text{SiO}_2$ )	Glaucinite (ferric potassic silicate of variable composition)
Hematite ( $\text{Fe}_2\text{O}_3$ )	Staurolite ( $\text{HFeAl}_5\text{Si}_2\text{O}_{12}$ )
Limonite ( $2\text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ )	Hornblende (complex silicate containing Al, Fe, Mn, Mg, Ca, Na and K)
Turgite ( $2\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ )	Hypersthene [(Fe,Mg) $\text{SiO}_3$ ]
Goethite ( $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ )	Sulphates
Pyrolusite ( $\text{MnO}_2$ )	Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ )
Rutile ( $\text{TiO}_2$ )	Barite ( $\text{BaSO}_4$ )
Titanite ( $\text{CaO} \cdot \text{TiO}_2 \cdot \text{SiO}_2$ )	Phosphates [ $\text{Ca}_3(\text{PO}_4)_2$ , $\text{Ca}_5(\text{PO}_4)_3\text{F}$ , etc.]
Zircon ( $\text{ZrSiO}_4$ )	Fluorides
Cyanite ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ )	Fluorite ( $\text{CaF}_2$ )
Sulphides	Carbonaceous or bituminous matter
Pyrite ( $\text{FeS}_2$ )	Hydrocarbons and carbon
Marcasite ( $\text{FeS}_2$ )	
Galena ( $\text{PbS}$ )	
Sphalerite ( $\text{ZnS}$ )	
Chalcocopyrite ( $\text{CuFeS}_2$ )	

Of the minerals constituting the unmetamorphosed limestones, only a few are readily visible usually. The others are recognized only when they possess distinctive colors or when the carbonates are dissolved by

acid and the residues are examined microscopically. The microscopic examination of residual insoluble materials has become in recent years a fertile field of research and has shown the presence of many minerals not previously suspected to exist in limestones.

The minerals present in unaltered limestones belong to three classes as to origin: (1) minerals precipitated from solution either by organic agencies or by chemical inorganic precipitation; (2) minerals that have been carried into the ocean by the land waters in suspension and have been dropped into the calcareous oozes; and (3) minerals that have been introduced into the oozes or consolidated limestones in solution by infiltrating waters at some subsequent time. It is not easy to classify the minerals into these three groups, for it is known that some of them occur in two or possibly all three divisions. Nevertheless, it is well to recognize the different origins.

Of the minerals precipitated from solution, calcite is the most important. Practically all the calcite has been carried into the ocean in solution and there has been precipitation by chemical reactions or abstracted from the water by organisms and built into their structures, which later collected in the ocean bottom. Most of the dolomite and silica, as well as the whole group of easily soluble minerals, were doubtless precipitated in a similar way. In regard to many others, even including the silicates, there is still considerable doubt as to whether they may not have been formed in some places by precipitation from solution contemporaneous with the formation of the calcareous ooze.

Of those carried into the ocean in suspension, we may include virtually the entire list of limestone minerals, although it is probable that only small quantities of the more soluble ones were transported from the lands to the seas except in solution. The oxide and silicate minerals in the main are relatively insoluble, so that most of them will be left on the surface in the decomposition of crystalline rocks in which they are most abundant, and are then apt to be picked up by surface streams, carried in suspension and eventually reach the sea. The indications are that limestones have been formed in places where only particles of small size derived from the lands could have been carried. Such a condition may exist near shore as well as far from land. This means that land-derived minerals can seldom be detected except by microscopic examination. Occasionally layers are found where the particles are large enough to be readily distinguishable with the naked eye. Most limestones on casual examination appear to be free from foreign minerals and yet even the purest are found to contain small particles when the residues are studied after the solution of the carbonates by acid. In regions where deposition was near an area of igneous or metamorphosed rocks, the variety of minerals present is surprising. Most of them show evidences of rounding by attrition during transportation or pitting by corrosive action.



Many of the minerals present in limestones are of secondary origin, having been introduced by water circulating through the pores and larger cavities at a period following deposition of the oozes and continuing even after consolidation of the original sediments. It is generally believed that these new minerals are introduced by water considerably above the temperature of shallow meteoric or surface waters, such as would be found in deep-seated waters, although there is some evidence to support the belief that this process at times takes place at shallow depths. When the calcareous sediments have been buried beneath hundreds or thousands of feet of later deposits, there is no doubt that the circulating waters, regardless of their original source, are higher in temperature and have a decidedly higher mineral content, and hence do more work of introducing new minerals. Further, such waters do much work of concentration of the ingredients finely distributed throughout the original oozes. At one time it was believed that most of the silicates, among them the feldspars, could be formed only at high temperatures, but there are a number of cases tending to show that feldspars have formed in limestones that have never been subjected to high temperatures such as are necessary for the formation of most of the metamorphic minerals.

The metamorphosed limestones contain a greater variety of minerals than do limestones not so altered. Further, the minerals present are generally in larger grains. In addition to the minerals already listed, the following have been observed: sulphur, graphite, magnetite, corundum, emery, spinel, pyrrhotite, chalcocite, molybdenite, apatite, phlogopite, biotite, tremolite, actinolite, epidote, olivine, talc, serpentine and others of rare occurrence.

In many places the limestones have been subjected to such intense compression by mountain-building forces that the temperatures of the rocks and the circulating waters were sufficiently high to cause important mineral changes. At others, the limestones have been intruded by molten igneous matter, which has caused similar heating and new combinations. The minerals formed under either of these conditions are known as metamorphic minerals, and constitute a large group. In the metamorphism of calcareous rocks the greatest change is that of recrystallization by which marbles are formed from the original limestones.

The oldest limestones have been subjected to great earth pressures at several different periods and contain many metamorphic minerals, whereas the youngest limestones have been little disturbed and have few metamorphic minerals. Even in rocks of the same age, some are found in regions of former great disturbances and consequent heating, whereas in other sections such forces have never been exerted on the rocks. The pre-Cambrian limestones, as a result of greater age and more compression, contain many of the minerals listed above, the Paleozoic limestones have suffered less compression and have few new minerals, and Mesozoic and

Cenozoic limestones in most sections are practically devoid of metamorphic minerals.

*Physical Properties.*—The physical properties of the limestones, such as color, odor, texture, structure, hardness, toughness, specific gravity, porosity, strength, etc., are of little importance when the stones are used for the manufacture of cement. The structures may facilitate or interfere with quarrying and the textures may likewise have some effect on the crushing, but these conditions apply to all types of rocks.

*Origin.*—A discussion of origin of limestones is of importance in this chapter only because it helps to explain their variable character, a condition that confronts many cement companies. Limestones are primarily of organic origin, in that the  $\text{CaCO}_3$  has been abstracted from the ocean waters by organisms, either plant or animal. Some of these organisms use the carbonate in their structures but others precipitate it outside their bodies by chemical reactions resulting from their life functions. Other limestones are of inorganic origin, the precipitation having been effected by evaporation, concentration, pressure and temperature changes, etc.

The places of origin are varied, as limestones have been formed in fresh and marine waters and in shallow and deep basins. Accumulating evidence seems to indicate that the bulk of our limestones, except the argillaceous varieties, has originated in shallow marine waters near low-lying flat shores, to which little mud was being carried. Wind-blown dust and sand are present, sometimes in appreciable amounts.

Anyone familiar with the deposits of our ocean shores realizes the variable characters of the sediments. Likewise, the operators of many of the limestone quarries recognize the varying chemical composition of the successive beds of rock, and even the variations within short distances of rock of the same bed. These are due to the changing conditions of deposition in shallow water.

*Limestones Adapted to Cement Manufacture.*—For natural cement there is little need for careful choice of limestone—high and low magnesian as well as otherwise pure and decidedly impure limestones have all been utilized, either alone or mixed with other stones—but a different situation prevails if Portland cement is to be manufactured.

Perhaps the most common objectionable constituent of limestones for Portland cement is magnesia. Our pre-Cambrian and Paleozoic limestones generally contain considerable  $\text{MgCO}_3$ . Since this is converted into  $\text{MgO}$  in the kiln, and may remain in part inert, it is evident that its presence tends to decrease the strength of the cement. Standard specifications permit only 5 per cent in finished cement. This means that limestones containing upwards of 10 per cent  $\text{MgCO}_3$  are useless unless they can be mixed with enough other stone low in magnesia to bring the average down to the specified limit.

Other objectionable features of limestones for Portland cement are improper ratios of  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{SiO}_2$ , although these can now largely be overcome by beneficiation, which is described on a later page.

Desirable features of limestone are such mixtures of the requisite ingredients that little or no correction need to be made by the addition of other types of rock. Argillaceous limestones such as occur in the Lehigh Valley, Pennsylvania, and in some other places possess a distinct economic value over other types of limestone. They are fine grained and the proportion of calcareous, aluminous and siliceous constituents is fairly uniform, so that it is relatively easy to obtain a mix. Some of these limestones contain slightly too much  $\text{CaCO}_3$ , so that at times some clay

TABLE 4.—*Analyses of Typical Limestones Used in Portland Cement*  
PER CENT

	Ordovician (Jackson- burg) Argillaceous Limestone, Lehigh Valley	Pennsylvanian (Alle- gheny-Vanport) Limestone, Butler Co., Pa	Ordovician, Annville, Lebanon Co., Pa. <sup>a</sup>
$\text{SiO}_2$	13.44	1.64	2.52
$\text{TiO}_2$ . . . . .	0.23		
$\text{Al}_2\text{O}_3$ . . . . .	4.55	2.20	1.56
$\text{Fe}_2\text{O}_3$ . . . . .	0.56		0.55
$\text{FeO}$ . . . . .	0.88		
$\text{MnO}$ . . . . .	0.06		
$\text{CaO}$ . . . . .	41.84	53.62	
$\text{CaCO}_3$ . . . . .	71.71	95.76	94.44
$\text{MgO}$ . . . . .	1.94	0.86	
$\text{MgCO}_3$ . . . . .	4.07	1.82	0.90
$\text{Na}_2\text{O}$ . . . . .	0.31		
$\text{K}_2\text{O}$ . . . . .	0.72		
$\text{P}_2\text{O}_5$ . . . . .	0.22		
S. ....	0.33		0.015
C. ....	0.75		
$\text{CO}_2$ . . . . .	32.94	42.30	
$\text{H}_2\text{O}$ . . . . .	1.55		

<sup>a</sup> High-grade limestone shipped to Portland cement plants in the Lehigh Valley.

must be added, but more generally they are deficient in lime and it is necessary to bring in some high-grade limestone from other places.

*Chalk.*—In some places, particularly in the southern part of England, northeastern France, and Belgium, chalk has been used in the manufacture of Portland cement instead of ordinary limestone. Chalk is merely a fine, soft, friable limestone that was extensively developed both in Europe and in the United States during the Cretaceous period. Its use in cement is not unlike that of the more ordinary limestones except that it can be both quarried and ground more readily. In general the  $\text{CaCO}_3$  content is very high, although in places it contains appreciable amounts of clay

and irregular flint nodules. The chalk mainly used contains from 88 to 96 per cent  $\text{CaCO}_3$ .

## MARLS

*Calcareous Marls\** (*Bog Lime*).—In many limestone regions, or in places where glacial deposits contain ground limestone or limestone pebbles, the ground and surface waters may be sufficiently high in dissolved lime to support lime-secreting plants and animals. Algae are the most important organisms in the extraction of the calcareous matter. In a limestone valley in Lancaster County, Pennsylvania, blue-green algae living in a small stream have formed a deposit of calcareous nodules of

TABLE 5.—*Analyses of Michigan Calcareous Marl*

	Wolverine Portland Cement Co.		From Alpena, Mich., Dried	From near Grayling, Mich., <sup>a</sup> Dried
	Light Marl Dried	Blue Marl Dried		
$\text{CaCO}_3$ . . . . .	93.75	91.34	74.48	87.00
$\text{MgCO}_3$ . . . . .	2.42	0.77	0.50	0.91
Soluble $\text{SiO}_2$ . . . . .	0.18	0.42		0.13
Insoluble $\text{SiO}_2$ . . . . .	1.01	0.78		0.78
$\text{Al}_2\text{O}_3$ . . . . .	0.55	0.55	0.54	0.07
$\text{Fe}_2\text{O}_3$ . . . . .	0.25	0.40	2.36	1.30
Moisture . . . . .			1.25	0.60
Organic matter . . . . .			12.88	9.80
$\text{SO}_3$ . . . . .	Tr.	0.26	0.89	0.27
Alkalies and rest (by difference).	1.84	5.79		

<sup>a</sup> Sample lost 61 per cent of its moisture when dried.

sufficient thickness to have been worked on a small scale for agricultural lime. A maximum thickness of 8 ft. has been found.<sup>15</sup>

Conditions are seldom favorable in streams for deposits of such calcareous deposits of commercial importance. In lakes and marshes the situation is different and there deposits from 15 to 30 ft. thick, or even more, have accumulated. In the glaciated sections of the country there are scores of lakes where this material has formed since the withdrawal of the ice. The marl is dark gray to white, depending on the amount of organic matter present. Clay and sand are mixed with the calcareous matter, so that in purity it varies from practically pure  $\text{CaCO}_3$  to clay or sand with only a small percentage of lime. Naturally, the impure varieties are useless. The origin of the marl is attributed largely to the action

\* There is valid objection to the use of the term "marl" in this sense and "bog lime" may be regarded as preferable. However, since "marl" is the name used by the cement industry, it seems advisable to follow that usage.

of a water plant known as *Chara*, although blue-green algae and mollusca are also important. The  $\text{CaCO}_3$  coats the stems of plants in the lakes and is dropped on the bottom. In a few deposits the shells of small pelecypods and gastropods constitute the major portion, although this is rare.

In Michigan, these calcareous marl deposits are of great importance and have been worked in many places to supply the calcareous matter for Portland cement. The marl is readily dredged from the ponds, or if the ponds have dried up or can be drained it is easily dug. As dredged it contains from 25 to 50 per cent of moisture. When dry the marl is very light and porous. Although the supply in any one deposit is seldom large, there are so many of these marl lakes that an extensive cement industry has been established on this type of material. Clay is mixed with the marl to produce the requisite mix. The analyses in Table 5 are chosen from an elaborate report of the Geological Survey of Michigan.<sup>5</sup>

TABLE 6.—*Analyses of Shell Marls*  
PER CENT

	From Chuckatuck, Va.	From Yorktown, Va. <sup>a</sup>	From Grove, Va.	From Trent River, N. C.	
				Unwashed	Washed
$\text{SiO}_2$ .	7.24	9.85	5.81	32.86	9.30
$\text{Al}_2\text{O}_3$ }	4.92	2.07	1.18	2.64	1.16
$\text{Fe}_2\text{O}_3$ }		2.88	0.62		
$\text{CaCO}_3$ .	85.13	78.20	89.48	63.47	88.26
$\text{MgCO}_3$	trace	0.28	0.96	0.36	0.38
Moisture		1.98	1.37		

<sup>a</sup> Average of samples from 15-ft. thickness of marl.

*Shell Marls.*—The Tertiary formations of the Atlantic Coastal Plain contain extensive deposits of fossil shells in a matrix of shell fragments, clay and sand. These are especially well developed in Virginia, North Carolina and South Carolina. They are flat-lying beds ranging in thickness up to 20 ft. or more and in most places are exposed at the surface or thinly covered by loose sands or clay. Another favorable factor is that in a number of instances these deposits are located on or near tidewater. Four cement companies have been organized in Virginia and North Carolina to exploit deposits of this character for the purpose of manufacturing Portland cement. The only plant built and in operation is owned by the Lone Star Cement Corporation, which dredges the shells from an extensive pit at Chuckatuck, Va., close to the James River, a short distance above Norfolk. The shell material is crushed, loaded on barges and taken to the plant in West Norfolk. The clay is obtained from a deposit a short distance farther up the James River. The material consists mainly of rotten molluscan shells but with representatives

of several other groups of organisms, especially corals. No difficulty is experienced in getting a satisfactory mix.

At Grove, Va., along the James River, at Yorktown, Va., along the York River, and near Newbern, N. C., along the Trent River, similar shell deposits have been investigated but no cement plants have been built. At times the shells have been dug for fertilizing purposes. Many other deposits of similar character have not been prospected. In places the sand and mud are objectionable, but generally they can be eliminated in large part by washing and screening.

*Recent Shells.*—Here and there along the Atlantic, Gulf and Pacific coasts there are deposits of recent shells, some on land and some beneath the shallow ocean or estuarine waters. Some of the land deposits are known as “kitchen middens,” as they are composed of the oyster shells and clam shells discarded by the Indians. In some places these are sufficiently extensive to have been utilized for burning lime but do not contain enough material to warrant the erection of a cement plant.

Underwater deposits of modern oysters and clams are more extensive and have been investigated in several places. The best of these known to the author is the deposit being worked by the Pacific Portland Cement Co. at Redwood City, Calif. A deposit of loose oyster shells covers a large area along the west side of San Francisco Bay. The shells are mainly entire, detached, fairly fresh in appearance and range in size up to  $1\frac{1}{2}$  in. long and  $\frac{3}{4}$  in. wide. A few clam shells are present. No living oysters are known in the immediate section, so it is difficult to account for the great accumulation of these shells. In places the deposit is 30 ft. deep. They are contained in a matrix of fine blue mud. The shells are dredged, loaded on barges and taken to the plant for crushing and grinding. A mix can be readily obtained from the shells and attached mud. If there is too much mud, the excess can be eliminated by washing.

#### ALKALI WASTE

One of the waste products of alkali plants is precipitated calcium carbonate, which usually is remarkably pure. This, naturally, is adaptable for cement manufacture. However, if a magnesian limestone has been used, the magnesia may be too high for Portland cement. In some large alkali works, the amount of this waste product is so great that it has been used for cement. A similar waste product results from the extraction of magnesia from dolomite but nowhere is the supply large enough to be of any commercial significance for cement manufacture.

#### BLAST-FURNACE SLAG

Blast-furnace slag can be substituted for some of the ingredients that are used in the manufacture of Portland cement, and by its use true Portland cement can be produced of the same character as that made

from natural rocks. As is well known, blast-furnace slag consists of the major portion of the gangue of the iron ore and the limestone that has been added for a flux, together with some impurities of the coke. The gangue residue is mainly composed of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  and the flux residue  $\text{CaO}$  and such impurities as it may have had. There is also some iron present. By adding more limestone, it is possible to obtain a mix that when burned will result in the standard Portland cement. Where high-magnesia limestones have been used for flux the slag is obviously unfit for Portland cement.

Slag Portland cement is generally cheap, as a waste product is utilized. If the slag is granulated in water as it emerges from the blast furnace the grinding cost is comparatively low but if it is cooled slowly in the air it forms a hard mass, which is much more difficult to grind.

#### NONCALCAREOUS MATERIALS USED IN CEMENT MANUFACTURE

Table 2 gives a list of materials that are sources of the necessary ingredients of cement. Certain of these substances are described elsewhere in this volume. The impurities of the limestones, particularly the argillaceous limestones, may suffice for all the necessary  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  as well as  $\text{Fe}_2\text{O}_3$ . Occasionally, however, raw materials composed more largely of these substances must be added to the limestones. Clays and shales are by far the most common. Slates by composition are similar but because they are harder and grinding costs are increased, they are seldom used. Sand and sandstones are used in some instances to obtain the proper silica ratio, although it is preferable to have the silica added in the form of aluminum silicates instead of quartz. When high-iron or high-aluminum cements are desired, ordinary iron ore or bauxite is employed.

#### RAW MATERIALS OF CEMENT

##### DISTRIBUTION AND POLITICAL AND COMMERCIAL CONTROL

The materials necessary for the manufacture of cement are worldwide in their distribution. In some places the best quality of stone is lacking or in areas of crystalline rocks suitable stone may be entirely wanting. These conditions are unusual for wide areas. A U. S. Government map of 1932 shows cement plants in all but the following 13 states: Arizona, Connecticut, Delaware, Massachusetts, Mississippi, Nevada, New Mexico, New Hampshire, North Carolina, North Dakota, Rhode Island, South Carolina and Vermont. Absence of plants is mainly due to scant population and hence poor market demand or to the proximity of cement plants in other states.

A somewhat similar situation prevails in the other countries of the world. When the domestic markets warrant, a search for materials is made, and usually with success. Increase in population and advance-

ments in industry are responsible for the building of new plants. The South American, Asiatic and African countries are less well supplied with cement plants than Europe and North America, as their industrial needs are smaller.

There is little political or commercial control over the raw products of cement. Rarely are they shipped far, and less rarely do they cross political boundaries. Tariffs have been placed on the finished cement in order to exclude competition with countries where labor and other costs are less. The United States Congress levied a duty on foreign cement, yet some European cement still comes into our Atlantic ports.

TABLE 7.—*Production and Price of Cement, 1926-1935*

Year	World Production, Bbl	United States			
		Portland Cement		Natural and Puzzolan Cements	
		Barrels	Average Factory Price per Barrel	Barrels	Average Factory Price per Barrel
1926	365,869,787	166,635,061	\$1 71	2,104,891	\$1 39
1927	397,531,595	173,206,513	1 62	2,123,868	1 33
1928	423,330,106	176,298,846	1 57	2,210,404	1 31
1929	437,716,223	170,646,036	1 48	2,209,465	1 37
1930	413,948,830	161,197,228	1 44	1,792,083	1 38
1931	355,902,181	125,429,071	1 11	1,241,803	1 32
1932	283,197,287	76,740,945	1 01	456,785	1 35
1933		63,473,189	1 31	466,632	1 43
1934		77,747,765	1 54	671,588	1 42
1935		76,751,419	1 51	1,006,064	1 42

#### PROSPECTING AND EXPLORATION<sup>12</sup>

In prospecting and evaluating cement-rock property it is first necessary to find suitable rock and then search out the adverse and favorable conditions. One of the serious problems confronting some of the cement companies is the presence of objectionable amounts of magnesia. It so happens that along the northern Atlantic border, where the bulk of the Portland cement of the country is consumed, there is much magnesia in the limestones, so that care must be taken in locating sites for cement plants. Some great thicknesses of limestones, aggregating hundreds of feet, contain no beds low in magnesia. Other localities contain interbedded high-magnesia and low-magnesia limestones, and in such places it must be determined whether it is possible to quarry or mine the desirable stone commercially without detrimental contamination from the high-magnesia beds. The problem is, therefore, first to determine the thickness and the chemical composition of all the beds.

In the early days of the industry, when all the stone in the quarry was hand-loaded, it was possible to sort the rocks shot down by the large



blasts, as it is relatively easy to train the quarrymen to distinguish between high-magnesia and low-magnesia. At present hand loading is not practicable.

If the usable material is in a band or bands 15 to 20 ft. thick with the beds either horizontal or vertical, the deposit may be workable, whereas inclined beds may so increase the cost of quarrying the good stone as to make the proposition unprofitable.

In the examination of a region of interbedded high-magnesia and low-magnesia limestones a distinction must be made between the ordinary averages of chemical composition and the weighted averages. If all the beds were of equal thickness, averages would be alike. In the bed described in Table 8 the distinction is well brought out. Of course, it must be known how the sampling is done. Some prefer to have all the composite samples represent equal measured distances. The writer believes that a better picture of the exact situation can be obtained by sampling separately each bed regardless of the varying thicknesses.

TABLE 8.—*A Typical Limestone Bed*

Thickness, Ft.	CaO, Per Cent	MgO, Per Cent
10	33.42	16.66
2	42.66	2.46
8	29.10	15.93
3	48.22	1.27
Arithmetical average.. . . .	38.35	9.08

10 ft. $\times$ 33.42 CaO = 330.42	10 ft. $\times$ 16.66 MgO = 166.60
2 ft. $\times$ 42.66 CaO = 85.32	2 ft. $\times$ 2.46 MgO = 4.92
8 ft. $\times$ 29.10 CaO = 232.80	8 ft. $\times$ 15.93 MgO = 127.44
3 ft. $\times$ 48.22 CaO = 144.66	3 ft. $\times$ 1.27 MgO = 3.81

23	793.20	23	302.77
Weighted average		Weighted average	
(793.20 $\div$ 23).....	34.49 CaO	(302.77 $\div$ 23).....	13.16 MgO

In sampling thick beds it is necessary to take pains to secure fairly uniform amounts from all parts of the bed, since changes in chemical composition in different parts may not be noticeable in the appearance of the stone. These might be observed on weathered surfaces but obviously it is important to have the samples taken from fresh stone.

Having recognized the presence of useful and useless interbedded limestones in a property designed to furnish the stone for manufacture of Portland cement, it next becomes necessary to determine its real value if the outcrops do not furnish sufficient information. This can be done

either by trenching or by drilling. Naturally, the method chosen should be the cheapest, provided it will furnish the needed information.

Where the overburden is thin and the strata dip gently, trenching to expose the different beds is satisfactory. If certain of the beds are overlain by upwards of 10 ft. of overburden, it may not be economical to use this method. In addition, the edges of the beds exposed in the trenches may be so weathered that samples obtained for analysis may give misleading results. It is not unusual to find beneath a soil cover limestones altered by differential or selective solution to a depth of 6 to 8 ft. and locally even more. The alteration is mainly in the partial solution of the  $\text{CaCO}_3$ , but other chemical changes may be produced. Where the strata are horizontal or approximately so, trenching cannot be employed except along hillsides, where the beds outcrop, and there only when the hillside debris or talus is not too deep.

In prospecting limestones by drilling various conditions must determine whether the ordinary well drill or a core drill is to be employed. In some places, particularly where the strata are horizontal, either one can be used. In such cases there is sometimes a difference of opinion as to which is preferable. Under almost all conditions the well-drill method is much cheaper and should be chosen if the results are satisfactory. If the core drill could be relied upon to furnish 100 per cent of core, there would be no question of its superiority, but when the beds are shattered, cavernous, partly decomposed, or of varying hardness, the core recovery may be even less than 50 per cent. The percentage of recovery depends at times upon the skill of the drill operator. By running the drill more slowly and removing the core at short intervals better results can usually be obtained. When the core recovery is disappointing, the writer has insisted upon the collection of the cuttings from the sludge overflow by means of a screen attached to the top of the casing and an analysis of this material as well as of the cores.

In certain hard and fairly uniform rocks lying in a horizontal position, prospecting limestone by well drills will give reliable results. In other places, particularly where there is clay mixed with the cuttings and washing is necessary, the results may be largely in error. If certain strata are unusually soft, they may be so finely pulverized as to have most of the material washed out with the clay. At times washed samples actually show less  $\text{CaCO}_3$  in the analysis than the unwashed, even though there is considerable clay present. In well-drill prospecting there is also danger of pieces of the rock from higher levels falling to the bottom of the hole and becoming mixed with the samples from the lower strata. Further, a sample representing only a small part of the cuttings may not be truly representative of the interval drilled.

In regions of horizontal strata the best plan in many cases is to prospect most of the property by the cheaper well-drill method, and to check

by means of a few core-drill holes. Where the strata are vertical, or nearly so, naturally the well drill, capable of drilling only vertical holes, is not applicable, nor should core-drill holes be put down vertically, as in either case a thickness of rock only the diameter of the hole is proved. Under conditions of steeply dipping or vertical beds the character of the strata can be obtained only by inclined holes. If the holes can be driven at right angles to the bedding planes, the maximum number of beds will be penetrated, and that means the maximum amount of useful information with the minimum amount of drilling. Where the surface is flat and the beds dip steeply, it is generally advisable to drill the holes at an angle of about  $30^{\circ}$ , less if the rocks are fresh just beneath the soil cover, or at a steeper angle if there is a considerable depth of decomposed rock. There are two reasons for doing this. First, it is generally regarded as better not to extend the holes to a vertical depth much greater than the proposed quarry depth, simply because the information sought concerns only the stone that will eventually be quarried. Accordingly, the angle should not be too steep. The objection to having the hole only slightly inclined is that much of its length will be close to the surface and rather largely in stone that may have been affected by surface weathering.

In starting a new diamond-drill hole it is advisable to locate it so that a few feet of strata previously cut at depth will also be sampled in the beginning of the next hole. This is desirable as a check in certain cases, and in others is useful because the first few feet of hole pass through decomposed stone not representative of the same beds in depth.

The prospecting of strata either greatly folded or faulted is difficult. Perhaps some particular bed may be recognized, if it is cut more than once in the same hole, but in limestones it is seldom possible definitely to recognize individual beds in the core samples when they are cut more than once. By noting the angle at which the strata are cut throughout the entire hole and later comparing the results with the data from other holes, the true structure may be worked out. It generally requires both skill and patience. If the underground structure is complexly folded it becomes impossible to decipher the character of the folding from the cores, and the average composition of the entire holes must be considered in reaching an opinion as to whether the stone is desirable or otherwise. If the results show that it is on the margin of usefulness, it is extremely risky to plan to open a quarry unless the amount of drilling done is excessive by having holes spaced at very close intervals.

After results have been obtained there are many methods for recording them. Many graphic methods have been used for presenting as accurate a picture as possible and in a way by which the essential points can be easily grasped. Geological training and experience, plus common sense and a keen appreciation of the economics of the industry, are the requisites in any person prospecting a cement property.

## MINING METHODS

The quarry and mining methods employed by the cement companies have been thoroughly discussed by Bowles<sup>1</sup> and Thoenen,<sup>16</sup> so that it does not seem advisable to go fully into that subject. Naturally, the object in obtaining rock for cement manufacture is to get as clean and uniform a product as possible at the lowest cost. This is accomplished in the great majority of places by open quarrying. The character, shape and size of the quarry naturally depends on the thickness, uniformity and structure of the rock.

Generally there is a layer of thick or thin residual clay overlying the usable rock, which always must be removed in order to obtain a proper mix. In places where the rock is apt to be deficient in lime, an additional thickness of rotten rock in which much of the  $\text{CaCO}_3$  has been removed in solution must also be removed. Deep pockets of residual clay are also present in places, which can be excavated only by hand pick and shovel.

Where the limestone is obtained from open quarry it is customary to develop a rounded or half-circle face, so that materials may be obtained from several points at the same time and differences in composition averaged. As a quarry is deepened it finally attains a depth at which it is advisable to work it in benches instead of a single high face. Churn drills are commonly used for the drilling of the holes for blasting. The spacing of the holes from each other and from the face of the quarry varies according to the rock. A fair average is probably 15 ft. When the size of the quarry permits, a number of holes are shot at the same time. If some of the stone is thrown down in large blocks in the primary blasting, there must be considerable secondary blasting done to reduce the size sufficiently for loading.

Steam or electric power shovels have almost completely replaced hand loading in the quarry. The rock is generally taken out of the quarry in cars by cable but in some places trucks are employed.

Where the overburden of waste material is excessive or the workable beds are thin and steeply dipping, it is necessary to substitute underground mining for surface quarrying. A number of cement companies obtain all of their stone from mines. The methods employed are similar to those of coal mining except that the rock pillars are stronger and generally little or no timbering is required. A cleaner product can be obtained than in surface quarrying. Under most conditions the costs are greater, so that little limestone mining is done except when quarrying costs become excessive.

BENEFICIATION OF LIMESTONE FOR CEMENT MANUFACTURE<sup>10</sup>

Until recently cement manufacturers have been concerned only with the chemical composition of limestone and have completely ignored the mineralogical constitution. The fitness or unfitness of particular stones

has been determined by the chemist. Little consideration has been given to the removal of objectionable portions or to the rectification of improper ratios of the constituents except by mixing with other rocks of different composition.

The minerals of which limestones are composed have unlike physical properties and behave differently when the rocks are crushed. Calcite and dolomite, because of their excellent cleavage, are readily broken into fine rhombs; the mica is separated into thin flakes; the quartz without cleavage planes is less easily broken and the fragments are irregular in shape; and the minor minerals behave likewise in definite and distinctive manner. Some investigators therefore considered it possible to make some separation of the mineral constituents by grinding, screening or tabling. Experiments proved the feasibility of such a procedure for

improving the grade to such a slight extent as to be of little value in commercial operations. Separations based on specific gravity have been suggested but are difficult because of the slight differences between quartz, calcite and dolomite. The conclusions concerning the practicability of these methods have been negative.

With the extension of froth flotation into the field of the non-metals, experimentation on limestones was suggested. Some people considered the expense

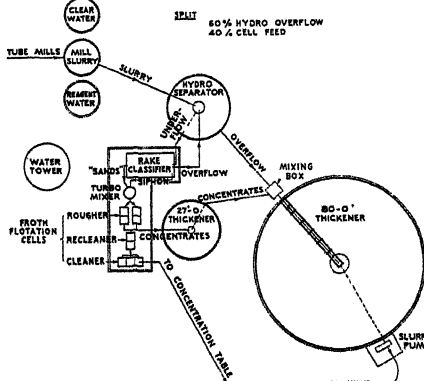


FIG. 1.—FLOWSHEET OF FLOTATION PLANT, VALLEY FORGE CEMENT CO.<sup>10</sup>

prohibitive, but as the result of a long series of experiments a commercial flotation plant was put in operation by the Valley Forge Cement Co., West Conshohocken, Pa., on March 15, 1934. It has been so successful that plans have been made by cement companies in Argentina, South Africa, France and Finland to erect similar flotation units for the beneficiation of their raw limestones. A rather wide variety of limestones has been used in the investigations and with success in each case, although each type of stone presents distinctive problems that demand changes in the procedure. There is no doubt that within a few years many, perhaps most, cement companies will process their limestones before the latter are fed into the kilns.

In the froth flotation of limestone the object thus far has been to remove only portions of certain minerals in order to obtain the requisite ratios for a mix without the addition of other stone. Usually this means a lowering of the  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  with reference to the  $\text{CaCO}_3$ . It is possible to separate the quartz grains, thus lowering the silica

content, or remove part of the mica and thereby lower both the  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ . The rectification may mean only a change of a few per cent or, as in one case now under consideration, it may involve an increase of  $\text{CaCO}_3$  from 40 to 75 per cent.

Each type of limestone calls for individual experimentation before the plant is planned, and, further, any flotation installation must be sufficiently flexible to meet the changing composition of the quarry rock coming into the mill. For this reason each flowsheet has its own char-

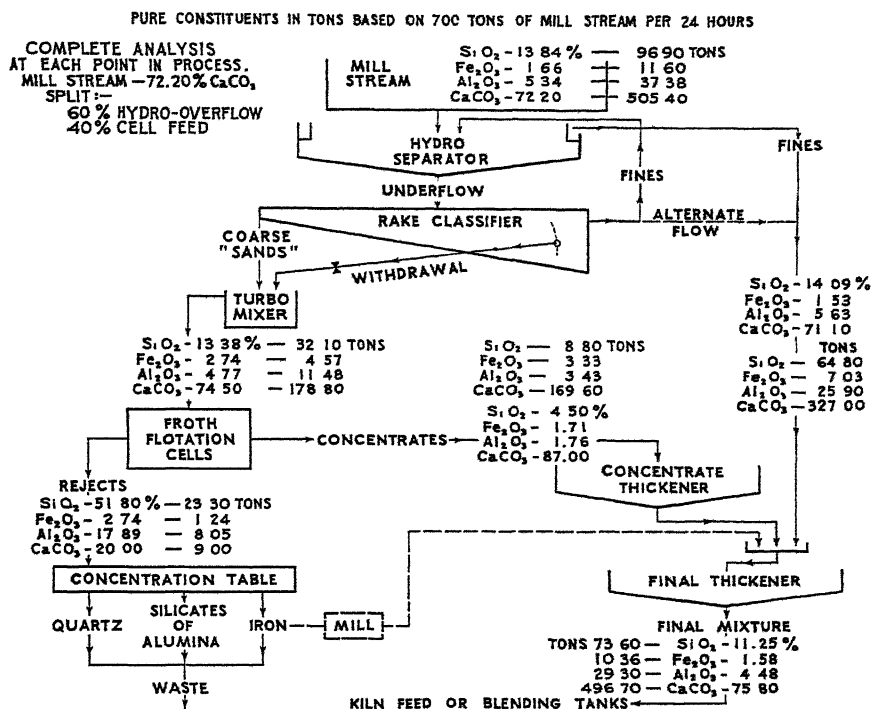


FIG. 2.—FLOWSHEET OF VALLEY FORGE CEMENT CO., SHOWING RESULTS OF ACTUAL OPERATIONS.<sup>10</sup>

acteristic features. Some of these are much more elaborate than the one now in operation by the Valley Forge Cement Co. but inasmuch as they have been developed in the laboratory alone up to the present time it seems inadvisable to include descriptions of them in this article. The two flowsheets presented (Figs. 1 and 2) illustrate the process and the analysis of the material at different stages. The concentration table has not been installed. The rock is ground fine enough for 85 per cent to pass through a 200-mesh screen. Oleic acid is used as a collecting agent and cresylic acid or B-23 alcohol to produce the frothing. The amount of stone passed through the flotation cells varies with the grade of the stone. If the mill feed is low in  $\text{CaCO}_3$ , a larger amount is withdrawn from the rake classifier and treated in the cells.

The principal advantages of limestone processing are summarized as: (1) reducing quarry costs in that poor stone when encountered need not be wasted, (2) otherwise useless stone situated near plant can be made useful, (3) grinding costs are reduced by elimination of the larger quartz particles, (4) fuel consumption is decreased by the discarding of some of the coarse quartz, (5) the quality of the cement is improved by making better ratios possible. These and other minor advantages in actual practice have been found to more than offset the cost of beneficiation.

Froth flotation in the beneficiation of limestone for cement is still so recent that it is difficult to predict the future. It may revolutionize the utilization of limestone for many purposes and develop in importance comparable to its position in the metallic products.

In the manufacture of high-alumina cement, bauxite is used. White clay is the additional ingredient used in the manufacture of white cement. For the production of dark colored cements and for cements that produce a minimum of heat in setting, several varieties of iron ore have been used.

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## CHAPTER X

### CHALK\*

By HEWITT WILSON†

**C**HALK is soft limestone formed from calcareous remains of microscopic organisms. Whiting is the finely divided product obtained from limestone rocks of which the chief constituent is calcium carbonate. Until recently most whiting was made from European chalk, but ground limestone, marble, and precipitated calcium carbonate have replaced a large amount of foreign chalk on the American market.

#### DISTRIBUTION, ORIGIN AND MODE OF OCCURRENCE

*European Chalk.*—The present outcrops of Cretaceous chalk are found in the area between Antrim, Ireland, and Uralsk, East Prussia, and from the northern deposits in Sweden and Scotland to the southern exposures near Nice in southern France. The present supply of commercial chalk and whiting is taken from the chalk cliffs between Dover and Hull, England, from near Mons, in the Departments of Pas de Calais and Nord in northern France, and also from Denmark and Belgium.

*United States.*—Some grade of limestone is found in every state of the Union, but the true chalk of Cretaceous age is limited to the Central and Southern States. Niobrara (Cretaceous) chalk outcrops are found as a narrow band encircling the Black Hills, in South Dakota, Wyoming, and Nebraska, and in much larger exposures in southeastern South Dakota, eastern Nebraska and western Kansas. A chalk band extends from west central Arkansas into Oklahoma and connects with the Austin beds, which outcrop on a northeast-southwest band extending almost across Texas. In the Southeastern States the Selma chalk extends from western Tennessee through northwestern Mississippi and across central Alabama. Some of the soft, creamy colored limestones of northern

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\* Based on an investigation made by the U. S. Bureau of Mines at its Northwest Experiment Station, Seattle, Wash., in cooperation with the College of Mines, University of Washington. Published by permission of the Director, U. S. Bureau of Mines.

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Florida, Georgia, Alabama, and probably Mississippi, Louisiana, and South Carolina, are similar to chalk in original hardness, fineness of grain, and working properties, but have a different origin.

*Origin.*—Although the remains of echinoids, mollusks, corals, sponges, and even vertebrate animals are included as fossils in chalk deposits, and are of primary importance in faunal and stratigraphic studies, they are secondary in importance to the microskeletons of coccolithophores and rhabdoliths that constitute the larger part of the chalk. These are plant organisms that secrete a number of calcareous (coccoliths) or trumpet-shaped bodies (rhabdoliths) on their surfaces, and on dying their calcareous remains mingle with precipitated calcium carbonate and the remains of foraminifera and other organisms that form an important part of deep-sea deposits.

#### COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

*Commercial Control.*—European chalks have had an advantage in the domestic market because of their light colors, plasticity, fineness of grain, ease of preparation by simple grinding and classification, and comparatively cheap transportation (by water). With the exception of a few American chalks from Arkansas and Kansas, from which putty has been made, domestic chalks have been used on an extensive scale only for Portland cement. The domestic chalks have the fineness of grain of European chalks, a similar paleontological origin, and an even stronger putty plasticity, but a recent study<sup>19</sup> indicated that the domestic materials did not equal the European in color and purity. Their chief value is for colored fillers and as plasticizers for the less plastic powdered limestones and marbles. Their occurrence is so widespread that undoubtedly better colored deposits will be found in the future.

Certain limestones of Illinois, Missouri and Pennsylvania develop good plasticity with the proper grinding treatment, and some of these have better color than the European chalks. The fortification of weakly plastic limestones and marbles with European chalks undoubtedly will continue for some time, especially along the Atlantic and Pacific coasts. These districts likewise may use ground shell as a plasticizer if a sufficient tonnage can be secured cheaply.

Some of the golden brown chalks of South Dakota, Kansas, Arkansas and Texas have colors similar to ochers and siennas and when diluted produce creams of more desirable hues than the gray-putty colors of the European chalks. The darkest of the Alabama chalks give a "driftwood" gray pigment on dilution.

The purest white calcium carbonate products can be made by grinding the white marbles from Georgia, Vermont, Alabama, California, and other states. These are far whiter than the European chalks but are of low

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<sup>19</sup> References are at the end of the chapter.

plasticity. Limestones and marbles as a source of calcium carbonate for precipitated whiting are more accessible to the eastern markets than are the domestic chalks found in the Central and Southern States.

The 1934 tariff rate for whiting or Paris white, dry, ground or bolted, is  $\frac{4}{10}$  of a cent per pound. Whiting ground in oil (putty) has a duty of  $\frac{3}{4}$  of a cent per pound. Crude chalk is duty free, while manufactured chalk products and precipitated chalk have a duty of 25 per cent ad valorem.

*Production and Consumption.*—Fig. 1 shows the quantities and values of imported whiting and products for the period 1920 to 1934 in compari-

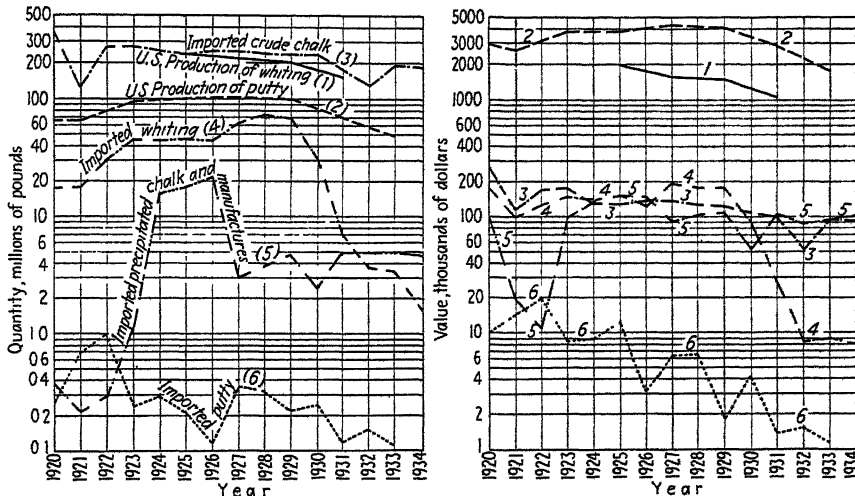


FIG. 1.—IMPORTS AND PRODUCTION OF CHALK AND WHITING FOR THE UNITED STATES, 1920 TO 1934.

Domestic production figures from the U. S. Bureau of Census, and import figures from Foreign Commerce and Navigation of the United States, U. S. Bureau of Foreign and Domestic Commerce. The production data are incomplete.

son with the total domestic production of whiting and putty. Imports of crude chalk and precipitated whiting have maintained a comparatively higher level since 1929 than importations of ground whiting and putty. It is not known how much of the domestic production was made from imported crude chalk.

#### PROSPECTING, EXPLORATION, MINING AND PREPARATION

*Prospecting, Exploration and Mining.*—The identification of the hard limestone rock is one of the simplest in the field because of its moderate hardness and effervescence with weak acids. However, the hardness test of calcite is lost in dealing with the softer chalks and marls and many impurities minimize the value of the effervescence test. Hence the purity of the sample and the character of the impurities must be deter-

mined by chemical analysis and petrographic studies. Core or churn drilling, prospect adits, or shafts are necessary to determine the extent and uniformity of the deposit unless these properties are indicated by satisfactory exposures. Freedom from grit is an important property in whiting and therefore quartz and other hard impurities must be determined. The removal of a stained overburden in advance of quarrying is very important in securing a white limestone for whiting. Limestone is obtained chiefly from open-pit quarries in which deep-hole blasting and power-shovel loading are employed.<sup>2</sup> The average mining cost, according to Thoenen,<sup>16</sup> for 30 of the open-pit limestone quarries in the United States was 67¢ per ton, but if the overburden is too heavy or the surface deposits are exhausted underground mining must be employed and the cost rises to about 96¢ per ton. Limestone is too low in price to justify mine timbering, and underground methods are used only where the rock is strong and sound enough to maintain roofs in drifts and rooms. Most of the 64 underground limestone mines in the United States studied by Thoenen<sup>16</sup> were of the adit or tunnel type, a few having vertical or inclined shafts. The single-breast stope was common for thin flat beds, and bench methods were used for thicker beds with the room-and-pillar system or its modifications.

*Ground Whiting.*—Chalk whiting made by the older English method is water-ground in edge-runner or stone-drag mills, after the flint pebbles have been removed by hand. The milky suspension is classified by water settlement in a series of tanks, producing waste sand and coarse particles in the first tank, "commercial," "gilders," "extra gilders," and "Paris white" in the following settling basins. Dewatering is accomplished by siphoning the clear water from the chalk sludge, filter-pressing and drying. The dried product is pulverized, sieved, and packed. Modern equipment for wet-ground whiting includes the use of rolls, Mueller grinders, and pebble mills for grinding; bowl and cone classifiers and hydroseparators for classification; and thickeners, continuous filters, and rotary driers for dewatering. In the cheaper dry process the crushed rock is dried in a rotary drier, coarse-ground in rolls or swing-hammer mills, and finely ground in impact pulverizers, roller mills, ball mills, or air-swept tube mills, often in closed circuit with mechanical pneumatic separators, which produce a product satisfactory for many whiting purposes. Quartz or porcelain-lined mills and flint pebbles are necessary for grinding the low-iron ceramic whiting.

*Precipitated Whiting.*—Quicklime, obtained by calcining limestone, is slaked to the hydroxide or milk of lime suspension, which is then treated with carbon dioxide gas produced by burning coke. The gas is scrubbed as it enters the upright recarbonation cylinders (each 6 by 18 ft.). The completion of the reaction is determined by phenolphthalein or other indicators and from 2 to 3 hr. is required for the reaction. The pre-

cipitated calcium carbonate is dewatered in filter presses and the dried filter cakes are pulverized for packing.

Precipitated calcium carbonate is obtained also as a by-product from the manufacture of caustic soda in paper mills:  $\text{CaO} + \text{Na}_2\text{CO}_3 + \text{H}_2\text{O} = \text{CaCO}_3 + 2\text{NaOH}$ . This product may contain traces of alkali, which restricts its use. Calcium carbonate is produced by other industrial chemical processes, including the manufacture of basic magnesium carbonate from dolomite. Certain "precipitated" grades apparently are not chemically precipitated but are merely finely ground and water-settled or precipitated from suspension, not solution.

Several manufacturers are now adding small amounts of fatty acids and their derivatives to limestone during fine grinding, to increase the plasticity, the mobility in the dry state, and the waterproofing and coagulation properties.

#### TESTS AND SPECIFICATIONS

Chemical analysis indicates the purity of the whiting, the character of the impurities, something of the color, especially the color that can be expected after ignition, and differentiates between the calcic and dolomitic varieties of limestone. The chemical requirements are particularly important in the chemical and ceramic industries, where a high content of calcium carbonate is required and the impurities, especially iron oxide, are not wanted. Class 1, ceramic whiting, is limited to a minimum of 96 per cent  $\text{CaCO}_3$ , a maximum of 1 per cent  $\text{MgCO}_3$ , 0.25 per cent  $\text{Fe}_2\text{O}_3$ , 2.0 per cent  $\text{SiO}_2$ , and 0.1 per cent  $\text{SO}_3$ . Class 2 permits 8 per cent  $\text{MgCO}_3$ .<sup>23</sup> The physical properties, such as fineness, color, working ability, time of settling, etc., which are the essentials for most fillers, bear little relation to the chemical composition, even though specifications may be based upon the chemical analyses of satisfactory whittings.

The physical requirements are more important than chemical composition in the paint industry, since such a wide variety of white and colored nonmetallic fillers, differing markedly in composition, are used in paints. The rubber industry limits its specifications to nearly pure calcium carbonate, which from the chemical basis can be supplied by many domestic marbles, limestones and precipitated whittings. However, fineness, strength, and workability are thought to be more important. The putty trade is interested in plasticity, oil absorption, oil retention and fineness.

Alkalinity is important in dentrifice, toilet and chemical products, and the curing of rubber is affected by variations in alkalinity. However, pure calcium carbonate is slightly soluble in water and the pH value of whiting is greater than 7.8. Both the Federal Government and the American Society for Testing Materials have titration methods

for determining alkalinity of whittings, but there is a need for research and correlation of these tests to specific service. A recent study<sup>19</sup> indicated that the precipitated whittings were highly alkaline (pH of 8.3 to over 9.6), which may cause a saponification of the linseed oil used in putty and paint.

Fineness specifications are limited to a restriction of those particle sizes coarser than 325 mesh. This is a satisfactory limitation of the coarse-grit content but is inadequate as an indicator of the other properties. Settling and sedimentation tests,<sup>17,18</sup> air separators,<sup>14</sup> and microscopic examination methods,<sup>7</sup> have been used in research laboratories for more accurate particle-size measurements of grains less than 50 microns in diameter.

The important properties required for manufacture of putty are: (1) plasticity, (2) hardening ability, (3) low oil absorption, (4) color, (5) chemical inertness, (6) insolubility in water, (7) resistance to "bleeding" oil, and (8) permanence. The best method of testing properties 1, 2, 7 and 8 is a simulative service test on the putty samples for several months, arranged by an experienced operator.

Although whiting cannot compete with white lead, zinc oxide, and other opacifiers, because of its low refractive index, it is used as an extender, body material or filler in oil paints. The depth of a suspension through which a given object or line can be distinguished under controlled conditions is used as a relative measurement of opacity.

Other tests of whiting include the settling rate in oil, the bulk density of the "fluffed" whiting to represent the freshly bagged product (4.4 to 10.0 grams per cubic inch), the volume loss (30 to 47 per cent) of the dry powder on packing and shaking, and the water-flow test to indicate the minimum amount of water required to make a flowing paste.

#### USES, MARKETING AND PRICE

The important uses include fillers or extenders in calcimine or cold-water paints, oil paints, putty, rubber, oilcloth, linoleum, window shades, cigarette papers, white ink, white shoe dressing, picture-frame moldings, dolls, dyes, tooth paste, fireworks, wire insulation, neutralizing agents in fermentation and general chemical processes, medicines and explosives. The paint industry is reported to use about 50 per cent of the imported whiting, the rubber industry about 35 per cent, and the putty trade about 10 per cent.

In many industries the chalk whiting is still preferred, especially the water-ground chalk and overground limestone and marble, and large quantities of duty-free, crude chalk are shipped to this country for domestic preparation along the ocean fronts where ocean freight rates from Europe have an advantage over rail rates from interior points. However, the use of better grinding equipment, control, and

more careful selection of raw material is giving the American producer a chance to establish his product in this country, especially in the Middle West.

After the high prices following the war, commercial chalk whiting remained at 1.0¢ per pound from 1922 to 1932, then dropped to 0.56¢ in 1934. It was quoted at 0.6¢ in the May 11, 1936, issue of the *Oil, Paint and Drug Reporter*. Gilders bolted chalk whiting ranged from 1.2 to 1.4 times that of the commercial grade. Gilders extra bolted and Paris white ranged from 1.2 up to 1.5 times the commercial grade, their present prices being 0.75¢. Prices for dry-ground commercial limestone, reported only since 1930, have remained at 0.5¢ for the product 99.75 per cent finer than 300 mesh (\$2 freight allowed). The present price of the wet-ground limestone is 0.575¢, the putty grind is 0.75¢, and the "precipitated" grade of ground limestone is 0.75¢ per pound. Chemically precipitated chalk in the five grades, extra light, light, medium, heavy and extra heavy, is priced at 3.0 and 5.0¢ per pound.

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## CHAPTER XI

### CHROMITE

By G. E. SEIL, MEMBER A.I.M.E.

**T**HERE are many varieties of the chrome ore known as chromite, only one of which is of commercial importance. In its pure state this ore consists of ferrous chromite,  $\text{FeO} \cdot \text{Cr}_2\text{O}_3$ . The commercial ores do not contain pure ferrous chromite, but consist of minerals derived from it, together with varying amounts of impurities.

Mineralogically, commercial chrome ores are composed of two definite and distinct components. The first, or primary component, is called chromite, and chemically this component is ferrous chromite in which a portion of the ferrous oxide ( $\text{FeO}$ ) has been replaced by magnesium oxide ( $\text{MgO}$ ), and a portion of the chromic oxide ( $\text{Cr}_2\text{O}_3$ ) has been replaced by alumina ( $\text{Al}_2\text{O}_3$ ). In other words, the primary component is a chemical entity, in which the molecular sum of the  $\text{FeO}$  and the  $\text{MgO}$  equals the molecular sum of the  $\text{Cr}_2\text{O}_3$  and the  $\text{Al}_2\text{O}_3$ . The secondary component of chrome ores, the impurities from a mineralogical viewpoint, consists of impure magnesium silicates. The impurities in the magnesium silicates may be various iron compounds, brucite, and aluminum silicate. The following magnesium silicates have been found as the secondary components of various chrome ores: clinoenstatite ( $\text{MgO} \cdot \text{SiO}_2$ ); enstatite ( $\text{MgO} \cdot \text{SiO}_2$ ); forsterite ( $2\text{MgO} \cdot \text{SiO}_2$ ); parasepiolite ( $2\text{MgO} \cdot 3\text{SiO}_2 \cdot 4\text{H}_2\text{O}$ ); chrysotile ( $3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ); antigorite ( $3\text{MgO} \cdot 3\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ); talc ( $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ ); hypersthene ( $(\text{MgO}, \text{FeO}) \cdot \text{SiO}_2$ ); serpentine ( $3\text{MgO} \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ); sepiolite ( $2\text{MgO} \cdot 3\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ). The chemical analyses of the chromites vary, of course, with the amounts of  $\text{FeO}$  and  $\text{Cr}_2\text{O}_3$  replaced by  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$  respectively. The analyses of the ores produced in one locality are peculiar to that locality and analytical data on the various ores will be given later.

#### PROPERTIES

The color of chromite is red to black, sometimes yellowish red in thin sections; streak, brown. The hardness is 5.5; specific gravity, 4 to 4.6. Luster is submetallic to metallic. Sometimes it is feebly magnetic.

The softening point of chromite is a function of the secondary component and varies from  $2300^\circ$  to  $2600^\circ$  F., although the mineral itself is

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\* Technical Director, E. J. Lavino and Company, Norristown, Pa.



classified as infusible. Chromite is one of the very few iron oxide compounds that cannot be reduced by CO. It is neutral and almost insoluble in slags and fluxes. Its reactivity increases directly with the percentage of iron present in the primary component. Its refractory properties

TABLE 1.—*Production of Chromite<sup>a</sup>*

Country	Crude Chromite, 1931-1935, Metric Tons					Percent- ages of Total World Pro- duction Based on Annual Average for the Years 1932-1934, Inclusive
	1931	1932	1933	1934	1935	
Australia .	61	99	905	1,744	605	
Brazil.					5 <sup>b</sup>	
Bulgaria.				85	<sup>d</sup>	
Canada (shipments)		71	27	101	1,038	
Cuba	15,197 <sup>c</sup>		24,154 <sup>c</sup>	50,162 <sup>c</sup>	48,509 <sup>c</sup>	5 59
Cyprus (shipments)	203	1,000		932	1,198	
Greece	5,634	1,555	14,784	30,694	31,984 <sup>b</sup>	
Guatemala	92 <sup>c</sup>		2,094 <sup>c</sup>	805 <sup>c</sup>		
India (British)	20,233	18,152	15,775	21,922	<sup>d</sup>	4 20
Indochina.	2,800				<sup>d</sup>	
Japan	9,675	12,492	19,897	27,222	<sup>d</sup>	4 48
New Caledonia	74,150	69,492	50,072	55,132	<sup>d</sup>	13. 14
Norway		409	326	42	<sup>d</sup>	
Philippine Islands.					21,292	
Rumania			29		<sup>d</sup>	
Southern Rhodesia	81,623	15,692	35,046	72,099	105,834	9 24
Turkey (Asia).	55,216	55,196	75,379	119,844	150,504	18. 84
South Africa .	23,335	19,371	34,078	61,357	90,431	8 63
Russia	67,000 <sup>e</sup>	68,000 <sup>e</sup>	112,723 <sup>e</sup>	129,200 <sup>e</sup>	<sup>d</sup>	23. 32
United States (shipments) . . .	272	157	857	375	523	
Yugoslavia. . .	58,384	43,925	26,248	47,352	60,000	8 84
Others . . .						3. 72
	414,000	306,000	412,000	619,000	<sup>d</sup>	

<sup>a</sup> Minerals Yearbook, 1936, U. S. Bureau of Mines.

<sup>b</sup> Exports.

<sup>c</sup> Imports into the United States. The figures for 1934 represent "imports for consumption"; those for prior years, "general imports."

<sup>d</sup> Data not available.

<sup>e</sup> Approximate production.

decrease as the iron increases. Pure ferrous chromite is not stable at high temperatures.

#### GEOLOGY, DISTRIBUTION AND PRODUCTION

The ore, as it occurs, is granular, compact, or is disseminated in country rock, and on rare occasions is found in small octahedral crystals. It is always associated with serpentine minerals and most of its deleterious

properties, from the refractory viewpoint, can be attributed to the associated minerals. Chrome ore occurs in peridotite, or in the serpentine derived from it, almost entirely as magmatic segregations. Frequently residual deposits occur, as chrome ore is very resistant to atmospheric influences, and it is, therefore, very common in gravels. The deposits, in place, are usually lenses of irregular outline, varying in size but seldom exceeding a few thousand tons.

Chrome ore was first found in the United States near the boundary line between Pennsylvania and Maryland. Prior to the World War, Southern Rhodesia and New Caledonia were for some years the chief producers of chromite. Their productions were about equal and together they furnished 80 to 90 per cent of the world's supplies. Of the other producing countries, Turkey, Russia, Greece and India were the most important. During the war, owing to shortage of ocean transportation and other causes, production was greatly stimulated in the United States, Canada and India. Following the war, conditions reverted practically to their prewar status. Recently, however, Russia, Turkey and Cuba have become important factors in the world's production of chromite. Development of deposits in the Philippine Islands indicates that this locality may also become an important source of supply. Today the United States is one of the smallest producers of the ore, although it is by far the largest user. Table 1 shows the world's production of chromite in metric tons and in percentages.

The United States contains sections where large amounts of chrome ore are found. Probably the largest known are in California and Montana, although there is some chrome ore in Georgia, Maryland and Pennsylvania. During the last few years production in the United States has not exceeded a few hundred tons annually.

Table 2 shows the domestic sales, imports and apparent supply of chromite in the United States for the years 1931 to 1935; also the chromite imported into the United States by countries for the same years.

#### USES AND PREPARATION

In the United States 50 per cent of the consumption of chromite is for metallurgical purposes, 40 per cent for refractory purposes, and 10 per cent for chemical and other purposes.

The specifications on the chromites vary with the use. For metallurgical purposes, ores high in  $\text{Cr}_2\text{O}_3$  are usually selected. For chemical purposes, ores which are easily decomposed, that is, the high-iron ores, are usually preferred. For refractory purposes the percentage of  $\text{Cr}_2\text{O}_3$  in itself is not a decisive factor, and ores relatively low in chromic oxide content are used if the percentage of  $\text{Al}_2\text{O}_3$  is relatively high. The physical condition of the ore and the placement of the gangue are important factors in ores for refractory purposes.

The chrome ore used in the refractories industry is sometimes heat-treated to recrystallize the chromite, thus stabilizing it, decreasing its chemical activity and distributing the gangue minerals so that their deleterious effects are minimized. Chromite recrystallizes slowly at 2900° F. and rather rapidly at 3400° F. Chromite that is heat-treated beyond the point at which recrystallization takes place, but below its fusion point, yields the best product for refractory purposes. Sometimes

TABLE 2.—*Crude Chromite in the United States<sup>a</sup>*  
LONG TONS

Year	1931	1932	1933	1934	1935	1935 value
Sales from domestic mines . .	268	155	843	369	515	
Imports (general) . . . . .	212,528	89,143	116,511	192,297	259,063	
Apparent available supply . .	212,796	89,298	117,354	192,666	259,578	
IMPORTED INTO THE UNITED STATES						
From						
Africa <sup>b</sup>	75,670	17,702	13,186	48,848	92,682	\$1,501,554
Belgium	482					
Brazil					2	26
Canada			49	30	2,888	62,941
Cuba	14,957		23,772	49,370	47,743	253,847
Greece . .	28,893	16,395	11,499	23,301	20,692	197,430
Guatemala .	91		2,061	792		
India (British) .	8,664	7,857	4,152	400	14,926	155,870
Netherlands.				259		
Oceania.	39,579	11,550	15,150	19,530	55,686	1,041,957
Other Asia . .				1,100		
Philippine Islands .					787	15,047
Turkey (Asia) .	2,198	17,602	27,854	28,730	16,060	267,294
Russia	17,736	4,800	13,261	19,937	3,412	53,721
United Kingdom	24,258	13,237			4,185	54,804
Yugoslavia			5,527			
	212,528	89,143	116,511	192,297	259,063	3,604,491

<sup>a</sup> Minerals Yearbook, 1936, U. S. Bureau of Mines.

<sup>b</sup> Includes Southern Rhodesia and Union of South Africa.

the chrome ore is recrystallized by fusion, but in ore thus treated the segregation and distribution of the secondary component in the cooled mass are usually poorer from a refractories-manufacturing viewpoint than they were in the original ore.

Chromite is used commercially for the manufacture of metallurgical products, refractories used in metallurgical plants, for cements and plastics used in the construction of furnaces, and for the production of chemical products such as chromates and pigments.

Most chromite is mined from quarries or open pits, although there are some underground operations. The ore is usually shipped in the condition in which it is mined. However, there are concentration plants in Serbia, in Turkey, in Canada, and in California, from which shipments of concentrates for the manufacture of chemicals, refractories and alloys are made.

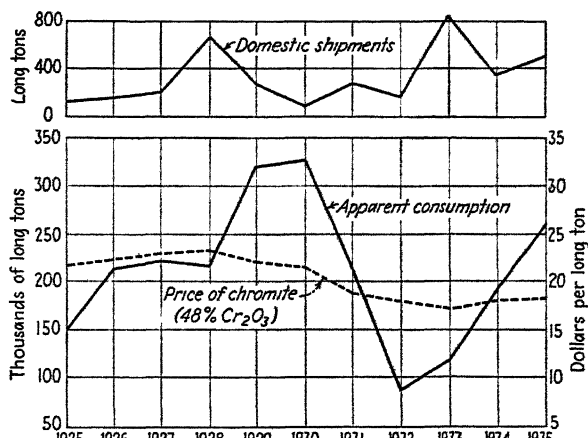


FIG. 1.—TRENDS IN CONSUMPTION, PRICE AND DOMESTIC SHIPMENTS OF CHROMITE IN THE UNITED STATES.

From Minerals Yearbook, 1936, U. S. Bureau of Mines.

#### ANALYTICAL METHODS AND DATA

In Table 3 are given analytical data on chrome ores imported from various countries since 1929, together with the tonnages represented by the analyses. These tonnages do not represent the total imports for the year indicated.

The solution of chromite for analytical purposes is very difficult. For the determination of  $\text{Cr}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$  and  $\text{MgO}$ , the best method of solution consists in fusion of a very finely ground sample with sodium peroxide in an iron crucible. For the determination of  $\text{FeO}$  a fusion with sodium peroxide in a silver or nickel crucible is required. Dr. G. Frederick Smith, of the University of Illinois, Urbana, Ill., has recently developed a new method for the solution of chrome ores, using a mixture of three parts of 85 per cent phosphoric acid and eight parts of 95 per cent sulphuric acid. This method is applicable when the sample weighs less than 150 mg. It is almost impossible, however, to completely dissolve larger samples, such as 0.5 gram, by means of this method, except by double treatment.

To determine  $\text{Cr}_2\text{O}_3$  the sodium peroxide fusion is boiled with water and acidified with sulphuric acid. The solution is completely reduced with standard ferrous ammonium sulphate and titrated with standard

potassium permanganate, using ortho-phenanthroline as an internal indicator.

To determine  $\text{SiO}_2$  the sodium peroxide fusion is boiled with water, acidified with perchloric acid, and evaporated to perchloric acid fumes. Water and hydrochloric acid are added and the solution boiled to remove impurities from the silica. The silica is filtered and the filtrate is evaporated to perchloric acid fumes a second time in order to remove all of the silica.

To determine  $\text{FeO}$  the sodium peroxide fusion, made in a silver crucible, is boiled with water and diluted to 500 c.c. in a volumetric flask. The solution is filtered and the residue is dissolved in dilute nitric acid. The silver is precipitated with hydrochloric acid, the solution is boiled to coagulate the silver chloride, and the latter is filtered off. The iron is determined in the filtrate.

To determine  $\text{Al}_2\text{O}_3$  the sodium peroxide fusion is boiled with water, diluted to 500 c.c. in a volumetric flask, the residue is allowed to settle, and the solution is filtered. C.P. ammonium chloride is then added to a 200 c.c. aliquot of the clear filtrate, and the solution is boiled. The resulting precipitate of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  is removed by filtration and the required separation of the two is made.

The accurate determination of  $\text{CaO}$  and  $\text{MgO}$  in chromite is difficult. The sodium peroxide fusion is boiled with water to remove the hydrogen peroxide. The solution is then acidified with acetic acid just sufficiently to turn blue litmus paper red. The solution is boiled, diluted to 500 c.c. in a volumetric flask, and filtered, the  $\text{CaO}$  and  $\text{MgO}$  being determined in aliquots of the filtrate, the interfering elements remaining in the insoluble residue. This procedure is an adaptation of the basic acetate separation. The  $\text{CaO}$  and  $\text{MgO}$  are precipitated together as phosphates and then separated by the conventional methods.

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TABLE 3.—*Analytical Data*

Country	Year	Tonnage	Analyses, Per Cent <sup>a</sup>					
			Cr <sub>2</sub> O <sub>3</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO
Brazil.. Canada...	1929	1,100	42.59 (±2.50)	13.82 (±1.50)	14.60 (±1.00)	7.98 (±1.00)	2.06 (±1.00)	15.89 (±1.00)
	1933	50	43.09 (±2.00)	16.77 (±1.50)	10.17 (±2.00)	8.92 (±1.00)	0.95 (±1.00)	17.98 (±1.50)
	1934	44,000	31.34 (±1.50)	14.30 (±1.00)	26.86 (±3.00)	5.60 (±1.60)	0.81 (±0.75)	17.21 (±1.50)
	1935	35,000	31.45 (±0.75)	13.88 (±0.60)	26.57 (±1.00)	6.31 (±0.60)	1.20 (±0.60)	17.74 (±0.50)
Cuba.	1936	50,000	30.48 (±2.50)	14.20 (±1.20)	27.48 (±2.50)	6.09 (±2.50)	0.88 (±0.60)	18.93 (±1.00)
	1929	24,000	37.43 (±0.10)	15.68 (±0.80)	21.40 (±2.25)	4.92 (±0.75)	0.75 (±0.30)	16.92 (±1.50)
Greece...			36.14 (±1.60)	15.43 (±0.60)	22.43 (±2.00)	5.24 (±1.80)	0.72 (±0.60)	15.96 (±1.75)
			38.33 (±0.80)	15.70 (±0.35)	21.62 (±1.25)	4.68 (±0.20)	0.61 (±0.50)	16.48 (±0.10)
	1930	400	46.03 (±1.15)	19.58 (±0.20)	13.64 (±0.70)	4.74 (±1.60)	0.43 (±0.30)	11.65 (±1.25)
Lalapanza (Africa).	1935	200	54.39 (±1.00)	14.12 (±1.00)	11.62 (±1.00)	2.54 (±0.60)	0.55 (±0.30)	16.05 (±1.00)
	1936	1,000	52.09 (±1.80)	13.00 (±0.25)	27.61 (±1.50)	5.31 (±0.40)	1.05 (±0.30)	18.18 (±1.50)
Philippines.	1934	35,000	45.43 (±0.45)	15.05 (±1.15)	13.84 (±4.00)	7.48 (±1.25)	0.46 (±0.35)	13.03 (±2.50)
	1935	51,000	44.93 (±2.80)	15.29 (±0.80)	12.94 (±0.60)	7.74 (±1.15)	0.53 (±0.60)	14.30 (±1.50)
	1936	70,000	46.46 (±2.50)	12.81 (±0.75)	15.42 (±2.50)	7.02 (±1.30)	0.88 (±0.80)	13.69 (±1.40)
	1930	13,000	39.08 (±1.00)	14.74 (±0.25)	15.63 (±2.50)	7.72 (±0.30)	1.15 (±0.60)	16.39 (±1.00)
Russia....	1938	12,000	40.02 (±1.00)	14.11 (±0.10)	15.33 (±2.50)	10.08 (±0.30)	0.85 (±0.30)	16.47 (±0.75)
	1934	20,000	46.19 (±1.50)	15.63 (±0.75)	14.58 (±2.00)	5.96 (±0.50)	0.91 (±0.15)	15.39 (±1.50)
	1930	20,000	47.72 (±2.10)	12.22 (±1.75)	12.49 (±1.85)	6.18 (±2.60)	0.86 (±0.75)	15.89 (±2.90)
Serbia..	1930	7,000	44.89 (±1.50)	24.99 (±1.75)	14.71 (±1.50)	2.21 (±1.60)	0.70 (±0.60)	10.82 (±0.80)
	1934	17,000	45.02 (±1.50)	25.73 (±1.75)	15.05 (±1.50)	1.76 (±0.50)	0.74 (±0.40)	9.92 (±0.85)
Transvaal (Africa).	1930	3,600	46.96 (±2.50)	12.46 (±1.00)	13.45 (±1.00)	7.92 (±1.80)	1.29 (±0.75)	19.15 (±2.00)
	1932	23,000	40.51 (±0.40)	12.46 (±1.00)	10.95 (±0.75)	11.86 (±2.60)	0.86 (±0.50)	20.00 (±2.00)
Turkey.	1933	32,000	46.57 (±2.50)	12.89 (±1.00)	12.50 (±1.00)	6.74 (±1.30)	1.16 (±0.50)	17.25 (±2.00)

<sup>a</sup> Figures in parentheses indicate range of analyses.

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## CHAPTER XII

### CLAY

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THE term "clay" is usually applied to certain earthy rocks whose most prominent property is that of plasticity when wet. This permits them to be molded into almost any shape, which they retain when dry. Furthermore, they harden under fire. Clays contain certain hydrous aluminum silicates—the clay minerals—in appreciable amounts, but aside from this a number of other mineral grains, particularly quartz, may be present. Texturally clays are fine grained, and the so-called true clay particles are under  $2\ \mu$  in diameter.

#### MINERALS IN CLAY

Certain hydrous aluminum silicates that predominate in many clays are known as the clay minerals<sup>66, 67</sup> and include: kaolinite ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ), dickite ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ), nacrite ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ), halloysite ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ), allophane ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot n\text{H}_2\text{O}$ ), beidellite ( $\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ), nontronite ( $\text{Fe}_2\text{O}_3 \cdot 3\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ), and montmorillonite [ $(\text{Mg}, \text{Ca})\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ].

Kaolinite, dickite and nacrite are known as the kaolin minerals, and although they have the same chemical composition, crystallize in the same system, show the same orientation of the axial plane A, and similar indices of refraction, they differ somewhat in their other optical properties. Individually they show characteristic X-ray diffraction patterns, dehydration curves, and behavior towards dyes.<sup>67</sup>

In studying the dehydration curves of kaolinite and anauxite, Ross and Kerr found that they show nearly flat curves up to a sharp break at  $390^\circ$  to  $450^\circ$  C., following which there is a rapid loss of water up to about  $525^\circ$  C., when the curves flatten out again with slight continued loss until complete dehydration at  $700^\circ$  to  $750^\circ$  C. Dickite showed an almost flat curve until just above  $500^\circ$  C., when there is a strong loss up to  $575^\circ$  C., at which point it flattens out again. Nacrite showed a slower loss of water than the other two, with the second break in the curve at  $675^\circ$  C.<sup>67</sup>

The term "anauxite" has been applied to some minerals having the same optical and crystallographic properties as kaolinite, but differing from it in having an alumina-silica ratio approaching 1:3. Apparently

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it forms an isomorphous series with kaolinite.<sup>67</sup> Pholerite, which has at times been used in the literature, sometimes to apply to kaolinite, is now discarded, as is also the name Newtonite. Leverrierite is the same as beidellite.

Kaolinite is commonly formed by the weathering of other minerals, particularly feldspar. Dickite seems to be the result of hydrothermal action, but may be formed at times by waters of meteoric origin, while nacrite so far as known always owes its origin to hydrothermal activity.<sup>67</sup> Halloysite is crystalline, as shown by the X-ray, and has a number of lines in common with kaolinite, but some are different. It always appears to be a weathering product, is very fine grained, white, and approximately isotropic, with an index near quartz. Its association with kaolinite is common.<sup>68</sup> Allophane is commonly glassy and amorphous, and not infrequently occurs as incrustations and thin seams alternating with halloysite, into which it may possibly change. Ross and Kerr consider it to be a solid solution of silica, alumina and water.<sup>68</sup> Montmorillonite, which is a common alteration product of the glassy particles of volcanic ash, appears in most places to have been formed by the action of sea water, since it is found in marine sediments, but in other instances may be the result of weathering.<sup>66</sup> Beidellite may also be derived from volcanic ash, but it is more abundant in ordinary clays and shales, in which it seems to have been derived from the weathering of mixed detrital material.<sup>66</sup>

Petrographers have also detected a "sericite-like mineral" in clay, for which the name illite has been recently suggested by Grim.<sup>24,66</sup>

It seems probable that the clay minerals may exert an influence on the physical properties of clay. Beidellite, for example, is thought to increase the bonding qualities.

Many other minerals have been identified in clays, but few of them occur in quantity.<sup>57</sup> Those that may be present, sometimes in appreciable amounts, are: quartz, usually in grains of variable size; calcite, usually in fine-grained, perhaps colloidal, form, but sometimes as concretions; limonite, often finely distributed as a coating on grains, sometimes as concretions or crusts; gypsum, in grains, selenite plates, crystals or rosettes; siderite, sometimes finely distributed, or occasionally as concretions in some clays and shales; pyrite as grains and concretionary lumps; muscovite, widely distributed, and commonly in very small flakes; rutile, almost universally present, but only in scattered grains of microscopic size.

While the clay minerals may form in place from the other minerals in residual or even some sedimentary clays, in some instances undoubtedly they originate under other conditions, as through the combination of colloidal alumina, silica and water. Thus Ross and Kerr describe long grains or worms of kaolinite noted in some Coastal Plain sands, which

could not have been transported and did not originate from the quartz grains.<sup>67</sup> There is also good evidence that kaolinite may replace quartz, as in the indianite deposits of Indiana,<sup>59</sup> and other places.<sup>67</sup>

The mineralogist commonly regards clays as made up of mineral grains, some of which may be very small, while the chemist and soil technologist frequently refer to them as consisting largely of colloidal particles, without any reference to a mineral composition. Thus Meyer<sup>44</sup> speaks of clay particles as being formed by the partial or complete combination of weak acids and weak bases, the proportion in which these are combined depending on the pH value of the surrounding medium. The clay particle, furthermore, he considers as consisting of a core made up of one or several molecules,\* carrying on its surface basic or acidic groups, or atoms, or positive or negative ionic charges, or both. Outside of this is the micellar ion atmosphere with adsorbed or unadsorbed ions. These together constitute the micelle. Basic and acidic atoms or groups are held on the surface of the clay particle by valence bond.

*Effect of Electrolytes.*—When thoroughly broken down or dispersed, the particles making up a clay cover a wide range of sizes. Some settle from a dilute suspension in a few minutes, but others, on account of their very small size, remain in suspension for a long time. These clay particles are negatively charged, and may be affected by small amounts of electrolytes, which exert an important influence on their settling properties. Some tend to increase the charge on the clay particles and help to keep them in suspension or dispersed. Such electrolytes are known as dispersing, deflocculating or peptizing agents. Other electrolytes have the opposite effect, in that they tend to reduce the charge on the clay particles, so that they unite, flocculate or coagulate. These may be called coagulating agents, and they reach their maximum effect when the negative charge on the clay particle is reduced to zero.

Clays do not all respond in the same manner to acids or alkalies, because each one may show its own acidity or alkalinity. Furthermore, the presence of soluble salts may exert a modifying effect. Dispersing electrolytes include sodium silicate, sodium hydroxide, sodium carbonate, sodium oxalate, sodium phosphate, etc. Coagulants include acids, sodium chloride, calcium chloride, aluminum chloride, etc. Some electrolytes, like sodium carbonate, may cause deflocculation when added to the clay in small amounts, and coagulation when larger amounts are added.

*Base Exchange.*—This process, representing the alteration in cation composition of a solid when treated with a salt solution, is probably of much importance in clays. According to the definition, base exchange may operate with either colloidal particles or larger grains of mineral

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\* Meyer states (personal communication) that these molecules may have the composition of clay minerals.

matter; in other words, it may operate in the weathering of rocks or in fine-grained sediments. Opinions seem to differ as to whether base exchange involves a change in crystal structure. It is probable, however, that it may go on in clays either during or after deposition, and Ross and Kerr<sup>67</sup> assert that it may take place without any breakdown of the clay molecule as a whole. The former suggests that certain clay minerals and zeolites possess so open a crystal lattice that certain cations can be displaced and other bases substituted in their place without disruption of the primary space lattice. Marshall<sup>41</sup> believes that this is the type of base exchange shown by most clays. He has measured the double

TABLE 1.—*Analyses of Clays*

	1	2	3	4	5	6	7	8	9	10	11	12
SiO <sub>2</sub>	46 3	45 78	57 62	59 92	68 62	82 45	54 64	38 07	47 92	49 56	53 12	90 00
Al <sub>2</sub> O <sub>3</sub>	39 8	36 46	24.00	27 56	14.98	10.92	14 62	9 46	14 40	15 08	18.72	4 60
Fe <sub>2</sub> O <sub>3</sub>		0 28	1.90	1 03	4 16	1.08	5 69	2 70	3 60	3 44	1 12	1 44
FeO		1 08	1.20									
CaO		0 50	0 70	tr	1 48	0.22	5 16	15 84	12 30	1 08	1 40	0.10
MgO		0.04	0 30	tr	1 09	0 96	2 90	8 50	1 08	7 84	6 92	0.10
K <sub>2</sub> O		{ 0 25	{ 0.50	{ 0 64	3 36	{ }	5 89	2 76	{ 1 20		5 72	tr.
Na <sub>2</sub> O			{ 0 20						{ 1 50		tr.	tr.
TiO <sub>2</sub> ...						1.00			1 22	0 40		0 70
H <sub>2</sub> O	13.9	13 4	10 5	9 7	3 55	2 40	3 74	2 49	4.85	22 96	12 04	} 3 04
Moist		2 05	2 7	1 12	2 78		0 85					
CO <sub>2</sub>							4 80	20 46	9 50			
SO <sub>3</sub>			0 35						1.44 <sup>a</sup>			

<sup>a</sup> Also 1.34 organic matter.

- |                                       |  |
|---------------------------------------|--|
| 1. Kaolinite.                         | 8. Brick clay, Milwaukee, Wis.                 |
| 2. Washed kaolin, Webster, N. C.      | 9. Shale clay, Ferris, Tex.                    |
| 3. Plastic fire clay, St. Louis, Mo.  | 10. Bentonite, Otay, Calif.                    |
| 4. Flint fire clay, Salineville, Ohio | 11. Potash-bearing bentonite, High Bridge, Ky. |
| 5. Loess, Guthrie Center, Ia.         | 12. Sandy brick clay, Colmesneil, Tex.         |
| 6. Siliceous clay, Rusk, Tex.         |  |
| 7. Brick shale, Mason City, Ia.       |  |

refraction of clay particles as small as 50  $m\mu$  ( $m\mu = 0.00001$  mm.) and finds that it varies according to the cation present. Such measurement is possible in clays whose particles show oriented coagulation, and works accurately for particles smaller than 500  $m\mu$ . He also notes that clays with high base-exchange capacity have a greater capacity for oriented coagulation.

Sodium chloride solution in contact with an ordinary clay results in a displacement of some of the calcium or magnesium in it. Conversely calcium or magnesium may displace sodium. Only clay minerals with easily replaceable bases, such as calcium, magnesium, potassium or sodium, exhibit much base exchange. Montmorillonite is quite susceptible to it.

*Chemical Composition.*—Clays vary widely in their chemical composition, from those close to kaolinite to others that show a high percentage of impurities. Table 1 shows this variation. A chemical analysis of clay is not usually carried out in detail, so that the  $\text{TiO}_2$  is included with the  $\text{Al}_2\text{O}_3$ , instead of being determined separately. All volatile matter is commonly expressed as loss on "ignition." All iron is usually determined as  $\text{Fe}_2\text{O}_3$ . Other unsatisfactory features of the chemical analysis are that it gives us little information regarding the physical properties of the clay, or the distribution of the constituents in the samples analyzed.<sup>57</sup> Moreover, it is unsafe to attempt to calculate the mineral composition from the bulk analysis. A modification of the ordinary quantitative analysis, known as the "rational analysis," attempts to determine the compounds present. It was first applied to kaolins, which were assumed to consist of quartz, feldspar and kaolinite (clay substance). The method is not reliable, however, and no satisfactory way of making this type of analysis has been devised.<sup>57</sup>

#### PROPERTIES OF CLAY

*Plasticity.*—The property of plasticity, already defined, is the outstanding characteristic of clays. They vary from those of high plasticity, or "fat" ones, like the ball clays and bonding clays, to those of low plasticity, termed "lean," and represented by some very sandy ones. The plasticity may be affected by the amount and character of colloidal material, the quantity and proportions of nonplastic particles, the amount of water, as well as salts, bases, acids and organic matter.

The cause of plasticity has been much discussed<sup>46,57</sup> and has been variously assigned to hydrous aluminum silicates, shape and size of grains, colloidal content, etc. The present general view regarding plasticity is well expressed by Norton, who says: "It is undoubtedly due to an active particle surface, which has the property of attracting to it a stable water film. This attractive force both holds the water in the pores and the particles together." There is no doubt also that the plasticity is influenced by the thickness and viscosity of the water films around the particles, as well as the size, shape and distribution of the latter.

The plasticity of a clay may be reduced by the addition of nonplastic material. Preheating a clay before use, to temperatures varying from 100° to 300° C., has a similar tendency.<sup>9</sup> Greaves-Walker found that the addition of hydrochloric acid to North Carolina shales increased their plasticity.<sup>23</sup>

While the particle size has sometimes been suggested as the chief cause of plasticity, it has been pointed out by Parmelee that mixtures of very fine-grained clays and coarse-grained ones are more plastic than either of these alone.<sup>46</sup> Very fine grinding of some minerals gives some plasticity, but nothing like that observed in clays. It is also claimed that an excessively high colloidal content develops stickiness but not plasticity.

*Texture.*—The grains of clay cover a relatively wide range of size, some clays containing sand particles large enough to be recognizable with the naked eye. A large percentage of the particles may be less than  $2\mu$  (0.002 mm.) in size, and when the clay is thoroughly dispersed the smallest, which are negatively charged, may remain in suspension for days.

In most ceramic work the separation of the particles in a clay is commonly made with sieves, some as small as 300 mesh. The Bureau of Public Roads uses the hydrometer test to determine the sizes from sand down, and Table 2 gives such a series. By using a super-centrifuge,

TABLE 2.—*Mechanical Analyses of Clays Made by Hydrometer<sup>a</sup>*

No. <sup>b</sup>	Particles Larger than 2.0 Mm.	Coarse Sand, 2.0 to 0.25 Mm.	Fine Sand, 0.25 to 0.05 Mm.	Silt, 0.05 to 0.005 Mm.	Clay Smaller than 0.005 Mm.	Colloids Smaller than 0.001 Mm.	Passing No. 40 Sieve
1	10	51	25	16	8	6	65
2	3	26	45	16	13	9	85
3	1	14	34	28	24	1	90
4	3	6	23	57	14	0	96
5	0	3	3	6	88	78	97
6	0	1	14	52	33	16	100
7	0	2	9	43	46	22	100
8	0	0	1	14	85	80	100

<sup>a</sup> Supplied by U. S. Bureau of Public Roads.

<sup>b</sup> 1. Sandy clay from South Carolina. 2. Sandy clay from Madison County, Virginia.  
3. Frost-heaving silt soil from New Hampshire. 4. Frost-heaving silt soil from Oregon.  
5. Colloidal clay soil productive of landslides in Virginia. 6. Expansive clay from Kansas.  
7. Gumbo from Red River Valley, Minnesota. 8. Wyoming bentonite.

Marshall<sup>41</sup> has separated the smallest particles of clays, as shown in Table 3. These clays, after removal of organic matter, soluble salts and exchangeable bases, were dispersed after bringing to pH 9 with NaOH.

TABLE 3.—*Mechanical Analyses of Clays by Centrifuge Method*

Clay	Concen- tration, Per Cent	2 $\mu$ –1 $\mu$	1 $\mu$ – 0.5 $\mu$	500– 200 $\mu$	200– 100 $\mu$	100– 50 $\mu$	Under 50 $\mu$
Kaolin. . . . .	0.2	66.0	21.0	7.0	6.0	39.0	
Bentonite <sup>a</sup> . . . . .	1.0	2.0	31.0	16.0	12.0		
Putnam clay . . . . .	0.5	7.8	6.6	11.8	11.6	21.3	40.9
Rothamsted . . . . .	0.5	15.2	12.1	18.7	14.3	10.3	29.4

<sup>a</sup> Approximate values obtained by interpolation in the distribution curve.

*Water in Clay.*—Two kinds of water are usually recognized in clay:  
(1) mechanically held water, and (2) chemically combined water.

When a clay dries from its plastic condition to a constant weight at room temperature, the water that evaporates until air shrinkage ceases is known as "shrinkage water." That which is still left in the intergrain spaces is termed "pore water," and may be driven off at 110° C. The pore water and shrinkage water together are known as the "water of plasticity." There may also be moisture retained on the surface of the clay particles as a film of molecular dimensions, which is tenaciously held and which is referred to as hygroscopic or micellar water. It may not all pass off until the clay is heated to 200° C. Table 4 gives the range of water of plasticity as recorded for a number of types of clay.<sup>57</sup>

TABLE 4.—*Water of Plasticity of Clays*

	PER CENT		PER CENT
Crude kaolin	36.39-44.78	Plastic fire clay	13.00-37.00
Washed kaolin	44.48-47.50	Flint clay	9.00-19.00
White sedimentary kaolin	28.00-56.25	Brick clay	13.00-41.00
Ball clay	25.00-53.00	Sewer-pipe clay	11.00-36.00
Crucible clay	27.00-51.00		

A high percentage of shrinkage water is rather characteristic of fine-grained clays that dry to a strong body, and they are also likely to show excessive plasticity, high shrinkage, warping and cracking. A high content of pore water characterizes a clay with a porous structure. The ratio of shrinkage water to pore water is said to be important in clays used in the manufacture of crucibles and glass pots,<sup>46</sup> and the best ones are said to show a ratio of 1:1 between shrinkage water and pore water. Table 5 gives the properties of several clays with respect to water and shrinkage.<sup>46</sup>

TABLE 5.—*Water and Shrinkage in Clays*

Clay	Water Plasticity	Volume Drying Shrinkage	Shrinkage Water	Pore Water	Ratio Pore Water to Shrinkage Water
Kentucky ball... ..	54.8	30.3	28.8	26.0	0.90
S. P. G. ball.....	48.2	25.0	21.5	26.7	1.24
English ball.....	40.9	25.8	21.1	19.7	0.93
Georgia kaolin.....	33.5	15.3	11.4	22.0	1.93
Missouri Hercules..	30.3	18.3	12.7	17.6	1.38

Clay is very hygroscopic and when dry absorbs moisture from the atmosphere, some absorbing as much as 10 per cent of its dry weight. The chemically combined water is chiefly that held in combined form as a part of the hydrous aluminum silicates, and passes off mostly at a temperature of 450° to 600° C.

*Shrinkage.*—Clays exhibit two kinds of shrinkage, air and fire. Air shrinkage occurs as the clay dries and continues until the particles are all in contact. It depends in part on the water content, and character

of the clay, being high in very plastic clays and low in sandy ones. An excessive air shrinkage tends to cause cracking, but a low air shrinkage is usually characteristic of clays that dry to a weak and porous body.

Open porous clays are easier to dry than dense, highly plastic ones, for in the latter water evaporates from the surface more rapidly than it can be drawn from the interior, and this develops stresses, which cause cracking. Very plastic clays therefore require slow drying.

Air shrinkage may be recorded in terms of the length or volume of the dry clay, the two being called respectively linear and volume shrinkage. Both linear and volume shrinkage may be measured (see Tests), but the former can also be calculated from the latter.<sup>57</sup>

TABLE 6.—*Air Shrinkage of Clays*

Clay	Linear, Per Cent	Volume, Per Cent
Crude kaolin	5.00- 7 6	14 11-20.92
Washed kaolin	3 00-11 0	20 00-29 00
Georgia kaolin	4 50-12 5	7 50-36 46
Ball clays	5 25-12 0	22 00-32 00
Flint clays	0 78- 6 5	2 3 -21 00
Sagger clays.	3 00-11 0	9.0 -25 00
Paving-brick clays	0 90- 6 0	3 5 -18 00
Sewer-pipe clays	3 50-10 5	16 12-22.90

*Strength.*—The strength of clay in its dried condition is an important property, as it enables it to withstand shocks in handling of the dried ware; also, a clay of high strength is capable of carrying a larger amount of nonplastic material, without too great deterioration of strength.

Strength of a dried clay may be determined by tension, compression, or transverse tests. The first was formerly much used, but is now discarded, the second finds little favor, while the third is the one commonly employed. It is expressed in terms of the modulus of rupture, and is carried out on the clay dried at 110° C., ranging in different clays from a few pounds per square inch to, in extreme cases, over 1000 lb. The figures in Table 7 give some idea of the range of transverse strength in different types of clays.<sup>57</sup>

TABLE 7.—*Range of Transverse Strength in Clays*

Clay	Lb. per Sq. In.
Washed kaolin . . . . .	75- 200
Georgia sedimentary kaolin . . . . .	150- 166
Ball clays . . . . .	25- 600
Glass-pot clays . . . . .	173-1068
Sewer-pipe clays. . . . .	190- 589
Sagger clays . . . . .	46- 474
Brick clays. . . . .	50-1500

The transverse strength of most clays usually decreases when sand or ground flint is added but occasionally an increase in strength may result, because the clay alone develops minute cracks in drying, which weaken it. The addition of flint avoids this and the true strength of the clay manifests itself.<sup>46,57</sup> It has also been shown that the transverse strength of a clay may increase with an increase in its base exchange capacity.<sup>31</sup>

The bonding strength of a clay refers to its power to hold together particles of nonplastic materials, such as standard sand, potter's flint, or "grog" (crushed brick). The property is important in the use of pottery, glass-pot, and crucible clays,<sup>10</sup> as they have an appreciable amount of nonplastic material mixed with them.

*Color.*—Iron is the commonest coloring agent of raw clays, giving yellow, pink, reds and browns, depending on the amount present and the state of oxidation. Greensand usually gives a green color. Organic matter may color a clay gray or black—sometimes even pink. Clays free from these coloring agents are usually white. Fired clays may owe their color to iron compounds, titanium oxide, or lime reacting with iron, but iron is the usual cause of the color.<sup>46,57</sup>

The best white-burning clays have under one per cent  $\text{Fe}_2\text{O}_3$ , but some have 2 per cent, although the latter tend to develop a light cream tint. An exact prediction regarding the color-burning qualities cannot be made from the chemical analysis, partly because it does not show whether the iron is evenly distributed. Buff-burning clays may vary from 1 to 5 per cent  $\text{Fe}_2\text{O}_3$ , but just why this color may be obtained from such a wide iron range is difficult to explain. Most of them have 3 to 4 per cent  $\text{Fe}_2\text{O}_3$ . Red-burning clays have 5 per cent or more  $\text{Fe}_2\text{O}_3$ . Such clays at a low temperature tend to burn pale red or salmon, but as the temperature increases they turn to a darker red and finally purplish or even greenish purple. The more siliceous clays usually develop a brighter shade of red. Much depends on the kiln atmosphere, for if this is reducing iron gives a bluish or bluish black color. Some white, cream or buff-burning clays become bluish gray at certain temperatures, owing possibly to the formation of ferrous silicate. This is called bluestoning. Lime in excess of iron oxide, if the two are evenly distributed, gives a cream-colored product, unless overfired, when the clay turns greenish or greenish yellow. Before the iron and lime begin to react, the clay may be pink, if enough iron is present. Titanium oxide to the extent of perhaps 2 per cent causes a creamy tint. This may explain why some clays very low in iron oxide do not burn white.

*Porosity.*—The porosity of a clay refers to its volume of pore space expressed in terms of its total volume. In raw clay the pores are all open but of variable size. In fired clays the pores may be of variable size, but are of two types, open and closed, the latter being formed by the expansion of gases during fusion. Porosity in the raw clay influences



its drying qualities, in that large pores permit the water to escape more rapidly. In fired clays the shape and size of the pores affect the properties of the ware, such as strength, behavior as an absorbent, resistance to weathering, shock, abrasion, corrosion, discoloring agents, efflorescence, destructive action by fungus growths, dielectric strength, etc.<sup>46</sup>

The temperature-porosity relations in firing serve to show the manner and progress of vitrification. Thus a clay in which porosity decreases rapidly, because of sudden fluxing action, is one that vitrifies quickly, and when a rise of the porosity curve quickly follows the drop in porosity it indicates a short firing range, such as would be characteristic of highly calcareous clays.

It is not uncommon to determine the absorption of a fired clay for water, instead of its porosity, since the curve of the former in a general way follows the latter, but is always lower.

*Specific Gravity.*—The specific gravity of a clay may be expressed in three different ways:<sup>46,57</sup>

1. True specific gravity, or the ratio of solid material, exclusive of closed or open pores, to an equal volume of water. This is determined on a powdered sample.

2. Apparent specific gravity, or the ratio of the volume of solids (plus the volume of any closed pores) to an equal volume of water.

3. Bulk specific gravity, or the ratio of the entire volume of material, including solids, closed and open pores, to an equal volume of water.

True specific gravity is affected by the minerals present in the raw clay, and in the fired clay by silica inversion, chemical reactions, fusion and crystallization. The apparent and bulk specific gravity are affected by all of the above, as well as by the porosity. In firing, the true and apparent specific gravity should theoretically decrease, while the bulk specific gravity increases. Change in specific gravity during firing indicates progress of vitrification. Table 8 gives the change in porosity and specific gravity of a Maryland clay.<sup>88</sup> Table 9 gives another series of somewhat complete determinations made on Kentucky ball clay.

TABLE 8.—*Changes in Specific Gravity and Porosity during Firing*

Temperature, deg. C. ....	1150	1200	1250	1275	1300	1325	1350	1375	1400
Porosity, per cent .....	22.8	22.0	10.1	9.6	6.6	6.3	1.4	0.7	0.4
Volumetric shrinkage, per cent ..	8.0	8.9	19.1	20.8	17.6	16.1	19.3	19.5	19.5
Apparent specific gravity .....	2.52	2.51	2.42	2.44	2.32	2.29	2.24	2.25	2.25
Bulk specific gravity .....	1.94	1.96	2.18	2.21	2.17	2.14	2.22	2.23	2.24

If the sample tested is a manufactured product, the amount of water and pressure used in molding may influence the results. Therefore there will be a difference in the bulk gravity, depending on whether the ware is slip-cast, hand-molded, plastic, or dry-pressed, if the same material is used.<sup>46</sup>

*Firing Changes.*—When a clay is fired it undergoes various changes in color, hardness, specific gravity, porosity, etc. Some of these have already been referred to; others are mentioned below. These changes may begin at a relatively low temperature range, or they may not be completed until higher temperatures are reached.

TABLE 9.—*Determinations on Kentucky Ball Clay*<sup>a</sup>

Cone	2	6	8	10	12	14
Volume fire shrinkage, per cent..	31.6	34.9	36.7	38.7	39.4	42.2
Apparent porosity, per cent. .	21.3	15.8	9.5	2.9	1.1	1.2
Total percentage of pores based on bulk volume of piece . . .	22.4	18.4		8.9	6.1	4.1
Closed pores . .	1.3	3.9		6.2	4.0	3.8
Specific gravity						
Bulk	2.09	2.18		2.32	2.39	2.47
True	2.69	2.679		2.541	2.515	2.587
Apparent	2.65	2.57		2.38	2.41	2.49

<sup>a</sup> Made by Parmelee and McVay.

*Loss of Volatile Products.*—The volatile products liberated during the firing of clay consist of: (1) chemically combined water; (2) organic matter, either carbon or bituminous matter; (3) oxides of sulphur or carbon given off when carbonates, sulphates or sulphides are dissociated. Dehydration of hydrous aluminum silicates takes place chiefly between 450° and 600° C., there being a slight variation in the different ones.<sup>15,46,57</sup> Gibbsite shows from 25 to 30 per cent loss at 310° C., and diaspor, 12 to 14 per cent loss at 540° C. Hydrous iron oxides generally decompose at 150° to 250° C., although some show the change between 250° and 300° C. Hydrocarbons, which may give trouble in firing, may be completely oxidized and removed by heating at between 800° and 900° C. Calcium carbonate at normal atmospheric pressure decomposes a little below 900° C., although the change may begin earlier. Siderite decomposes at 800° C., but in presence of clay this may begin at 425° C.<sup>46</sup> Pyrite begins to decompose at 350° C., while gypsum loses three-fourths of its H<sub>2</sub>O between 250° and 400° F., and all of its volatile matter by 1100° C.

The two following analyses, representing (1) an impure clay from Ferris, Tex., and (2) brick from that clay, are interesting as showing the loss of volatile products in firing:<sup>53</sup>

	1	2		1	2
SiO <sub>2</sub> ..	49.45	56.6	Na <sub>2</sub> O	0.21	1.4
Al <sub>2</sub> O <sub>3</sub>	17.11	20.4	TiO <sub>2</sub>	0.17	with Al <sub>2</sub> O <sub>3</sub>
Fe <sub>2</sub> O <sub>3</sub>	3.45	6.2	H <sub>2</sub> O	4.84	0.5
CaO.	12.67	11.7	CO <sub>2</sub>	7.10	
MgO	1.77	1.4	SO <sub>3</sub>	2.00	
K <sub>2</sub> O	0.13	1.5			
				99.43	99.7

*Fusibility.*—When exposed to a rising temperature clays do not fuse suddenly; on the contrary, they soften slowly until the entire clay becomes a viscous mass. With impure clays this may occur at a relatively low temperature, but with those approaching kaolinite in composition, it takes place at a much higher one.

Following dehydration, the clay is porous, but after a temperature interval, it begins to compact, then the more easily fusible minerals begin to melt with the formation of glass. With further temperature rise the fluid portion attacks the mineral grains not yet fused, and finally we get a solution of molten glass.

In the first stages of firing, although the clay may become compacted into a hard mass like common brick, it is said by some that there is no evidence of softening of the particles.<sup>46</sup> This period has also been called incipient vitrification, but by some that term is used to refer to the stage at which enough glass has been developed to bind the mass together. Complete vitrification would represent the stage reached in the fusion of a clay when sufficient glass has been developed to close all the pores. As the temperature rises and the fluidity of the glass developed increases, the clay mass or object no longer holds its shape, and reaches a condition referred to as viscosity. The temperature at which the development of glass begins, as well as its amount and viscosity, exerts an influence on the behavior of the clay during vitrification. In some clays glass has been found to develop at a temperature as low as 700° C. The term vitrification as applied to clay wares does not always mean the same thing. Paving brick and sewer pipe, for example, are said to be vitrified, but technical difficulties prevent the complete attainment of this condition. Electrical porcelain must approach it very closely.

The stages of incipient vitrification, vitrification and viscosity merge into each other, but the temperature interval between the first point and overfiring is variable. Clays having a long vitrification range are, in general, the safest to use for vitrified wares, as most commercial kilns cannot be controlled within a range of a few degrees of temperature, and there is less danger of the ware becoming overfired and ruined. Clays that begin to overfire as soon as they have reached a condition of vitrification are said to have a short firing range.

The curves of porosity and fire shrinkage shown in Figs. 1 and 2 illustrate well the behavior in firing of the two types of clay mentioned above.

Most fired-clay bodies when examined in thin section under the petrographic microscope show a variable amount of glass, depending on the amount of fusion that has taken place during firing, while scattered through this there will be noticed mineral grains that have not yet been affected. Some of these, however, may exhibit a certain amount of corrosion.

Relatively few new minerals will have been found to have crystallized out from the fused material on cooling. Small rods of hematite have been observed in the glassy matrix of some ferruginous clays, but the mineral that has been most often noticed, and which has attracted considerable attention, consists of needlelike grains, usually colorless. For some years these were identified as sillimanite, but later it was discovered that they were the mineral mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ). The temperature at which this develops does not seem to be the same always, but it has been observed to form at as low as  $900^\circ\text{C}$ . Mullite is a rare mineral, having been first observed in some igneous rocks on the island of Mull. Commercial

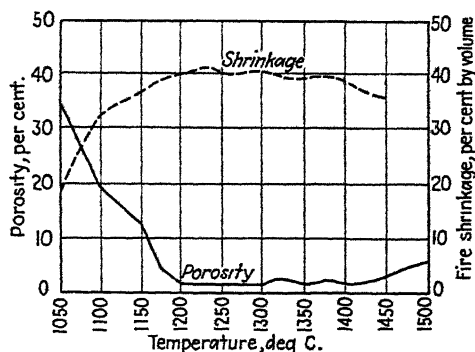


FIG. 1.

FIG. 1.—POROSITY AND SHRINKAGE CURVES OF A TENNESSEE BALL CLAY WITH LONG FIRING RANGE.

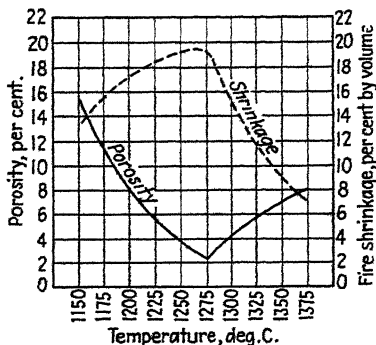


FIG. 2.

FIG. 2.—POROSITY AND SHRINKAGE CURVES OF CLAY WITH SHORT FIRING RANGE.  
Brown: *Jnl. Amer. Ceramic Soc.* (1918) 1.

deposits are found of the minerals sillimanite, kyanite and andalusite, all of which have the composition  $\text{Al}_2\text{SiO}_5$ . When these are heated they all change to mullite at temperatures between  $1400^\circ$  and  $1550^\circ\text{C}$ . It has been found that the development of mullite in the firing of certain ceramic bodies has great importance, because it imparts to the ware high tensile strength, superior dielectric properties, and low thermal expansion, properties that are particularly valuable in spark plugs. It is possible to use the proper amount of the minerals mentioned in the clay mixture and have them change to mullite in firing.

The fusion point of a clay is usually expressed in terms of Seger cones,<sup>57</sup> and may range from as low as perhaps cone 1 ( $1150^\circ\text{C}$ .) to cone 35 ( $1785^\circ\text{C}$ .).

**Soluble Salts.**—Many clays contain at least a small percentage of water-soluble inorganic compounds, which are brought to the surface in the drying of the ware, remaining there as a coating, usually white. These soluble salts are mostly sulphates of lime and magnesia, but sometimes others. Vanadium salts may also cause a stain.<sup>71</sup> Soluble salts may be in the clay when taken from the ground, or they may form as the

result of weathering, as when pyrite is present. They may also be introduced in the water used for mixing the clay. Others may be formed during firing, by the kiln gases carrying oxides of sulphur from the fuel, which, coming in contact with carbonates in the clay in the presence of moisture, convert them into sulphates.<sup>46, 53</sup>

Soluble salts in clays range from zero to 1.5 or 2.0 per cent.<sup>46</sup> They may be the cause of technical troubles because of: (1) the unsightly coating that they form on some wares, (2) interference with the adherence of a glaze, and (3) the crystallization of these salts in the pores of the product may cause its disintegration. Barium compounds are sometimes added to the clay to render the obnoxious salts insoluble.

### ORIGIN OF CLAY

Clays may originate from different kinds of rocks, either by the ordinary processes of surface weathering or by the action of solutions, which may be of igneous origin or indirectly of surface origin. In both cases the alteration product is of residual character, and the material may be called a residual clay.

The removal of the clays so formed by various agents of erosion and transportation and deposition elsewhere gives rise to a great group of transported clays.

The several ways in which clay deposits originate are brought out by the accompanying classification.<sup>57</sup>

### *Classification of Clays*

#### A. Residual clays.

Formed by weathering of rocks *in situ*, or by rising solutions of magmatic or meteoric origin.

##### I. Kaolins, white and firing white or light cream

- a. Deposits roughly tabular in form, as when derived from pegmatites, or hydrothermal alteration along fractures.
- b. Blanket deposits from areas of igneous or metamorphic rocks.
- c. Replacement deposits, as Indianaita.
- d. Bedded deposits from feldspathic sandstones.

##### II. Red-burning residuals, derived from different kinds of rocks.

#### B. Colluvial clays, practically landslide masses.

#### C. Transported clays.

##### I. Deposited in water.

##### a. Marine clays or shales. Deposits often of great extent.

White-burning clays; ball clays and sedimentary kaolins; refractory clays or shales; buff burning

Impure clays and shales  $\left\{ \begin{array}{l} \text{Calcareous.} \\ \text{Noncalcareous.} \end{array} \right.$

##### b. Lacustrine clays deposited in lakes or swamps.

Fire clays or shales.

Impure clays or shales, red burning.

Calcareous clays, usually surface deposits.

##### c. Flood-plain clays. Recent ones usually impure and somewhat sandy.

- d. Estuarine clays (deposited in estuaries), mostly impure and finely laminated.
  - e. Delta deposits. Variable purity, often lenticular.
- II. Glacial clays found in the drift. Often stony and lacking stratification. May be red or cream burning (calcareous).
- III. Wind-formed deposits (some loess).
- D. Chemical deposits (doubtful origin).

*Residual Clays.*—Those due to weathering may be formed: (1) by the decomposition of the silicate minerals in rock, the product being a clay; (2) by the solution of a carbonate rock containing clayey impurities, which are left behind as an insoluble residue, and (3) by the disintegration, accompanied occasionally by some solution, of shales.

In case 1, where the decomposition of silicates like feldspar is involved, the clay mineral formed is a hydrous aluminum silicate, such as kaolinite. Residual clays formed by weathering overlie the bedrock formation from which they have been derived. They may grade into it, as is usual when the weathering of silicate rocks is involved, or there may be an abrupt change from clay to bedrock, as in those derived from limestones. While residual clays have formed throughout geologic time, relatively few "fossil" deposits are found, since these clays are easily removed by erosion.

The thickness of such clays is variable and depends on the depth to which weathering has penetrated the rock, and the freedom of the deposit from erosion. Residual clays derived from rocks free or nearly free from iron-bearing minerals are white and represent the true kaolins but most residual clays are stained by oxides of iron.

Laterite is a peculiar type of residual clay, formed under such conditions that silica is largely removed during weathering, leaving alumina and iron hydroxides predominating in the residual mass. Its formation is associated with tropical climates, and a variety of rocks may yield it.<sup>83</sup>

Residual clays, usually of ferruginous nature, are found in many parts of the United States, particularly east of the Mississippi and south of the glaciated area. Within the latter they occur only in protected situations.

Kaolins are confined mainly to the belt of crystalline rocks of the Atlantic States from Delaware southward, and reach their greatest importance in western North Carolina. Commercially important deposits are also known at one locality in Quebec, and near Spokane, Wash. In England, the largest deposits known are in the Cornwall district, and these have for years been a large contributor to the American pottery industry. Residual clays formed by rising waters are relatively rare, and not as a rule of commercial value. However, such an origin has been ascribed by some to the Cornwall deposits, and those of Karlsbad, Czechoslovakia, as well as the Quebec and Washington occurrences.<sup>28, 86</sup>

*Transported Clays.*—Transported clays initially were derived from residual clays, but later on in addition from pre-existing sedimentary

deposits. As noted in the classification, they have been deposited chiefly in water, under a variety of conditions, which affect the extent and also the characters of the deposit. Those of recent geologic age are usually unconsolidated, but the older ones from the Tertiary downwards are often consolidated to shales, although there may be noticeable exceptions, as in some Tertiary and Cretaceous deposits of the United States.

Glacial clays are of limited occurrence and confined to the area of Pleistocene glaciation.

Wind-formed deposits, if this origin is accepted for the loess, are of widespread occurrence in the Great Plains area. Chemical deposits are a debatable group, in so far as workable ones are concerned.

*Secondary Changes in Clay Deposits.*—In areas of crustal disturbance, beds of sedimentary clays and shales are sometimes folded and faulted, and this may result in steeply dipping beds, as along the eastern edge of the Rocky Mountains. Some Paleozoic shale formations of the eastern United States show a regional dip. Weathering may often bring about such changes as leaching of lime carbonate, oxidation of the iron compounds, or opening of the structure. The transportation and redistribution of iron oxide by surface waters may cause the formation of crusts, and sometimes concretionary masses. Chert concretions are found in residual clays, but most concretions found in sedimentary clays are probably of contemporaneous origin, and may consist of limonite, iron carbonate or lime carbonate.

Shale beds in places are so softened by weathering near the outcrop that they easily yield a plastic mass when ground and mixed with water, but with depth the material may become much harder and occasionally have too little plasticity to be workable.

#### DISTRIBUTION OF CLAYS AND SHALES

Clays and shales are widely distributed in the United States and Canada, in formations ranging from pre-Cambrian to Recent. The more important types are:

*Kaolins.*—In their washed condition, kaolins are used in the manufacture of high-grade products, such as white earthenware, porcelain of all kinds, as well as fillers. They are rather limited in their distribution.

Kaolins resulting from the weathering of pegmatite are found in the belt of crystalline rocks extending from Delaware to Georgia,<sup>59</sup> but the only important deposits now worked are those of western North Carolina, and to a lesser extent Delaware. The pegmatites of North Carolina may be weathered to a depth of 80 to 100 ft. White residual clays derived from the Oriskany limestone have been worked near Saylorsburg, Pa., and others formed from schist were mined at South Mountain, Pa.<sup>59</sup> Kaolins from weathered dolomite have been mined in southeastern Missouri.<sup>59</sup> A group of deposits near Spokane, Wash., formed from pegmatite have been used for refractory wares.<sup>96</sup>

In Canada, the only kaolin deposit worked is near San Remi, Que. This is unique in that it is supposed by some to have been formed by the replacement of quartzite, caused by hydrothermal activity.<sup>29</sup>

The so-called sedimentary kaolins occurring in the Cretaceous of Georgia<sup>74</sup> and South Carolina,<sup>90</sup> have been worked for a number of years, being used in the manufacture of white pottery, refractories and as a filler. The deposits are usually lens-shaped and may be of considerable extent, as well as thickness. Bauxite is associated with some. The product may be washed before shipment to market.

A widely used clay, intermediate in its properties between true kaolins and ball clays, is an extensive deposit of white sedimentary clay occurring in the Tertiary (Eocene) of north central Florida.<sup>6</sup> It has been worked for over 40 years, and is known to underlie several counties. The material is exceedingly sandy, and is washed before shipment.<sup>6,59</sup>

*Ball Clays.*—These are sedimentary, plastic, refractory clays, which fire white or nearly so, and are used chiefly as one of the ingredients of whiteware, as well as for other purposes where a refractory clay of good bonding power is required.<sup>77</sup> The chief domestic supply comes from the Tertiary formations of western Kentucky and western Tennessee,<sup>45,59</sup> where the material occurs as large lenses associated with other refractory clays used for such purposes as firebrick, saggers, bond for abrasives, etc. These different grades may form separate layers at times in the same deposit.<sup>59</sup> A relatively small amount of ball clay is also obtained from the Cretaceous beds of New Jersey.<sup>61</sup>

The only locality in Canada where ball clay is said to occur is in southern Saskatchewan, and this is remote from important markets.<sup>42</sup>

*Fire Clays.*—Under this head are included all refractory clays, exclusive of kaolins and ball clays. Sedimentary clays of this character are found in the United States in formations ranging from the Carboniferous to Tertiary, exclusive of the Triassic.<sup>18</sup> Their geologic distribution in Canada is restricted to the Cretaceous and Tertiary.

Foremost among the clays of this group are those found in the Carboniferous of Pennsylvania,<sup>72</sup> Ohio,<sup>61</sup> Kentucky,<sup>54</sup> Missouri,<sup>85,91</sup> Indiana,<sup>92</sup> Illinois,<sup>32,47</sup> and Maryland.<sup>50,88</sup> The clays occur at a number of different horizons, and may be associated with coal. Most of the material is plastic fire clay, but with it there may be a hard type known as flint clay, which develops little plasticity, and which, because of its texture, structure and appearance, has received this special name. It is found particularly in Pennsylvania, Ohio, Maryland and Kentucky. Many of these fire-clay deposits are worked as open pits, but others are reached by drift or shaft. These clays are widely used for firebrick.

The Lower Cretaceous formations of New Jersey have for years supplied material for the refractory-products industry.<sup>60</sup> They are all plastic clays and are unassociated with coal. In Texas<sup>53</sup> and Mississippi,<sup>59,39</sup> the



Tertiary formations supply refractory clays. Those from the latter state have found favor because of their high bonding qualities, of value in the manufacture of glass pots and crucibles. Other Tertiary clays have been developed in California,<sup>19,78</sup> Washington,<sup>94</sup> and Colorado.<sup>17</sup> A unique type of refractory clay is that found in the north central Ozark region of Missouri,<sup>4</sup> where there is a series of basin-shaped deposits carrying flint clay, and some run rather high in diaspore. These diaspore clays, which Allen thinks have been formed by the alteration of the flint clay by the action of percolating solutions containing CO<sub>2</sub>, are of highly refractory character. Clays intermediate in alumina content between flint clay and diaspore are called burley clays in the Missouri district. The No. 1 grade is said to carry 60 to 70 per cent alumina.<sup>86</sup>

In Canada fire clays have not been as widely developed, nor are they as widely distributed, as in the United States. The most important deposits are those south of Moosejaw and in southern Saskatchewan. Extensive deposits are also known to occur and have been worked around Clayburn, in southwestern British Columbia. A curious series of deposits is known to have been preserved under a heavy cover of glacial drift on the Mattagami and Missinabi Rivers<sup>20,30</sup> of Ontario. The clays are of Mesozoic age and associated with sand deposits. Although they are reported to be of high quality, their remote location may hinder their commercial development.

*Stoneware Clays.*—Those employed for the better grades of stoneware are often at least of semirefractory nature, and of dense-burning quality.<sup>69</sup> They do not have to fire white. Such clays are found in part associated with the Carboniferous fire clays of the Central States, as well as in the Tertiary and Cretaceous formations of the East, South and West.

*Paving-brick and Sewer-pipe Clays and Shales.*—As a rule shales rather than clays are employed for this group of clay products. There is a large production from the Carboniferous shale formations of the Central States. Some of the Tertiary clays of Texas, California and Washington have also been used for this purpose. Some Carboniferous shale of Nova Scotia has been used for sewer pipe, and the clays of southwestern British Columbia have been similarly employed.

*Brick and Tile Clays.*—Practically none of the states are lacking in clays or shales that can be used in the manufacture of common brick.<sup>2,6-8,13,14,19,23,25,26,34,39,40,49-54,60,62-64,75,76,91,94</sup> The materials commonly used are red-burning surface clays of transported or residual types, and the clay sometimes selected is not of the best, especially if it is very sandy, as it makes a porous and weak brick. Probably the largest brickmaking district in the United States is that of the Hudson River Valley. Cream-burning calcareous clays are sometimes employed, not so much from choice as because they happen to be the common type of material in areas where such bricks are made, as around Mil-

wauke, Wis.,<sup>52</sup> and some parts of Michigan.<sup>14</sup> Shales of Paleozoic age have been used to some extent in the Eastern and Central States.

In Canada<sup>29,30,60</sup> somewhat the same types of clay are used as in the United States. The surface clays are mostly red burning, but around Winnipeg, Man., cream-burning clays are common,<sup>60</sup> and therefore are selected. Silurian shales are used at Toronto, and Cretaceous shales in parts of Saskatchewan.

*Europe.*<sup>55</sup>—The most important clays of Europe are the kaolin deposits. Those at Cornwall, England, which have been worked for years and to depths of several hundred feet are widely used,<sup>40</sup> not only at home but abroad. They are the largest deposits of their type that have been developed anywhere. Also important are the kaolins of Karlsbad, Czechoslovakia, and those worked at several localities in Germany.<sup>79</sup> The English ball clays are well known to potters, and, like the Cornwall kaolins, have been exported to the United States in quantity. Belgium, Germany and England<sup>21,84</sup> all contain excellent deposits of fire clay. That from Gross Almerode, Germany, has been exported to the United States for glass-pot manufacture.

#### POLITICAL AND COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

It can hardly be said that control is much of a problem in the clay industry, because the United States is relatively independent of other countries for its supplies of raw clays, and only certain special kinds of high-grade clays have been imported. These have been gradually replaced by domestic materials except perhaps the highest grade of paper-coating clays.<sup>86</sup>

For many years there has been a somewhat steady importation of English kaolin or china clay for use in the manufacture of china, paper, paint, etc. The strong hold this product has had on the American market for years has probably been due in part to its more uniform character. Before the war more than two-thirds of the china clay consumed in the United States was imported from England, and as late as 1925, according to Bureau of Mines statistics, the imports were more than the domestic production. The replacement of foreign clays in the pottery and paper industry continued during the depression, and in 1934 the china-clay imports formed less than 20 per cent of the domestic consumption. This increase in the use of domestic materials has been due in part to their more careful preparation for the market.

In former years much English ball clay was imported, the potters in this country preferring it to the American material, which did not vitrify at quite as low a temperature; indeed, when the American ball clays were first developed some of the potters used to the English product were loath to class the domestic product as ball clay. In recent years, however, the domestic clay has replaced the imported ball clays and glass-pot clays.

Since the large majority of the clay-working plants in the United States obtain their supply of raw material from their own deposits, the production of this clay is not listed separately. According to Tyler<sup>26</sup> roughly 35 million tons of clay was produced in the United States in 1929, but its chief use was in the manufacture of heavy clay products. The figures of clay production issued formerly by the United States Geological

TABLE 10.—*Clay Sold by Producers in the United States, by Kinds<sup>a</sup>*

Year	Kaolin or China Clay and Paper Clay		Ball Clay		Ship Clay		Fire Clay	
	Short Tons	Value	Short Tons	Value	Short Tons	Value	Short Tons	Value
1909-13 <sup>b</sup>	132,114	\$ 705,352	63,371	\$231,447	14,268	\$25,867	1,629,098	\$2,261,738
1925-29 <sup>b</sup>	453,618	3,834,825	116,127	890,457	6,839	37,000	2,810,001	7,747,918
1930.	533,800	3,893,814	93,488	739,787	4,398	26,465	2,547,162	6,070,663
1931	443,300	2,946,953	83,007	639,798	1,916	13,613	1,473,161	3,741,038
1932.	344,994	2,011,208	47,573	312,751	525	5,105	725,993	2,057,060
1933	411,233	2,366,339	64,551	400,564	1,562	11,365	1,133,693	3,141,545
1934.	426,335	2,699,016	62,377	423,421	5,117	35,832	1,288,909	3,733,033
1935.	523,585	3,766,109	90,489	607,730	4,316	25,839	1,783,321	4,532,565

Year	Stoneware Clay		Bentonite		Miscellaneous		Total	
	Short Tons	Value	Short Tons	Value	Short Tons	Value	Short Tons	Value
1909-13 <sup>b</sup>	142,569	\$ 143,034	c	c	400,546	\$ 369,019	2,381,966	\$ 3,736,487
1925-29 <sup>b</sup>	88,575	188,055	c	c	568,869	1,220,458	4,044,029	13,918,173
1930	75,832	146,513	107,405	\$ 858,927	360,818	785,326	3,962,903	12,521,495
1931	57,466	131,915	78,815	472,045	381,830	406,823	2,519,495	8,352,185
1932	49,736	82,521	71,613	503,673	151,382	229,291	1,391,816	5,201,609
1933	28,188	59,581	117,428	760,174	83,518	101,049	1,840,173	6,840,617
1934.	56,678	100,279	215,339	1,050,578	132,008	155,094	2,187,263	8,197,253
1935	35,460	70,280	189,840	1,071,412	168,913	216,490	2,795,924	10,290,425

<sup>a</sup> Minerals Yearbook, 1936, U. S. Bureau of Mines.

<sup>b</sup> Average.

<sup>c</sup> Up to 1930 bentonite was included under miscellaneous.

Survey and since 1923 by the United States Bureau of Mines represent chiefly clay that is sold to factories or firms on a royalty or tonnage basis. It consists mostly of high-grade clay used in the manufacture of white ware, refractories, or for filler in paper, paint or fabrics, and in some years is said not to have represented more than 10 per cent of all the clay mined. The figures of production do not include either clay used in manufacture of Portland cement, or that used for bleaching purposes, which is listed under "fuller's earth." It does include bentonite, which is used in part

for bleaching purposes as well as a variety of other uses, but not by itself for the manufacture of clay products. Even the fire clay reported does not include all of that type, as many refractories manufacturers operate their own deposits.

Table 10 gives the production and value of clays over a period of years.

It is interesting to note the rank of the five leading states, for production of each type of clay, as shown in Table 11.

TABLE 11.—*Clay-producing States in 1934, Arranged in Order of Rank*

Rank	Total Value	Total Tonnage	Kaolin and Paper Clay	Ball Clay	Fire Clay	Stoneware Clay
1	Georgia	Pennsylvania	Georgia	Kentucky	Pennsylvania	Pennsylvania
2	Pennsylvania	Georgia	South Carolina	Tennessee	Missouri	Ohio
3	Missouri	Missouri	Pennsylvania	New Jersey	Ohio	New Jersey
4	South Carolina	California	North Carolina		Kentucky	Washington
5	Kentucky	Ohio	Delaware		California	California

The reason for the reversed positions of Georgia and Pennsylvania in the first two columns is that the former state ranks first in value because of its output of sedimentary kaolin, while the latter is first in tonnage because of its large production of fire clay. In the fire-clay column, New Jersey would be only sixth. It produces considerable fire clay, but

TABLE 12.—*Salient Statistics of the Clay Industry in the United States, 1925-1929 and 1931-1934\**

Domestic Clay, Short Tons	1925-29 (Average)	1931	1932	1933	1934
Sold for:					
Pottery and stoneware	225,910	147,409	108,135	114,022	110,915
High-grade tile	74,350	96,632	44,329	32,101	26,310
Saggers, spurs, stilts, wads	205,627	73,117	39,832	49,916	51,160
Architectural terra cotta	91,753	31,188	13,520	12,875	9,659
Paper ..	201,292	275,469	230,445	255,989	249,852
Rubber	31,996	34,501	33,719	49,615	48,259
Oilcloth and linoleum	11,885	7,411	5,326	6,715	9,061
Paints.	18,047	12,920	7,983	10,859	11,869
Cement	70,913	121,196	50,281	22,747	24,581
Refractories	2,068,970	1,101,401	573,530	1,030,565	1,194,655
Miscellaneous	1,043,286	618,251	234,716	254,769	450,942
Total sold:					
Quantity	4,044,029	2,519,495	1,391,816	1,840,173	2,187,263
Value	\$13,918,173	\$3,352,185	\$5,201,609	\$6,340,617	\$8,197,253

\* Minerals Yearbook, 1934, U. S. Bureau of Mines.

much of it is mined by the manufacturer. The tonnage of Ohio stoneware clay happens to be greater than that of New Jersey, but the value is less.

It is also interesting to note the uses to which the different clays are put, and the relative amounts consumed.

The statistics in Table 12 indicate that since 1931 there has been a falling off in the amount of clay sold for every group of uses except in the paper and rubber industries. The demand for clay is governed in part by the conditions in the industries it supplies and in part by competition with substitutes.

There is naturally a close connection between fire clays and the iron and steel industries. In 1934, according to the U. S. Bureau of Mines, the demand for clays in the paper and rubber industries was increasing more rapidly than the industries themselves were. In the rubber industry this was due to the displacing of more costly materials by clay in

TABLE 13.—*Clay Imported for Consumption in the United States\**

Year	Kaolin or China Clay		Common Blue and Gross-Almerode Glass-pot Clay		All Other Clays				Total	
					Unwrought		Wrought			
	Short Tons	Value	Short Tons	Value	Short Tons	Value	Short Tons	Value	Short Tons	Value
1909-13 <sup>b</sup> .....	261,266	\$1,562,684	19,763	\$159,788	31,836	\$126,313	1,423	\$ 24,312	314,288	\$1,873,097
1925-29 <sup>b</sup> .....	339,014	3,055,885	12,130	113,307	57,001	493,983	4,047	52,550	412,192	3,715,725
1930.....	236,251	2,197,540	18,900	154,423	24,883	209,175	4,984 <sup>c</sup>	143,817 <sup>c</sup>	285,018 <sup>c</sup>	2,704,960 <sup>c</sup>
1931.....	151,426	1,056,393	15,133	116,446	15,615	125,326	8,376 <sup>c</sup>	237,359 <sup>c</sup>	190,600 <sup>c</sup>	1,536,024 <sup>c</sup>
1932.....	99,807	461,191	5,380	45,445	13,290	90,140	8,133 <sup>c</sup>	280,404 <sup>c</sup>	127,110 <sup>c</sup>	877,180 <sup>c</sup>
1933.....	116,180	632,437	7,099	69,639	17,623	141,992	9,756 <sup>c</sup>	336,435 <sup>c</sup>	150,658 <sup>c</sup>	1,180,503 <sup>c</sup>
1934.....	100,775	752,993	9,467	111,823	11,673	120,733	9,226 <sup>c</sup>	355,965 <sup>c</sup>	131,146 <sup>c</sup>	1,341,524 <sup>c</sup>
1935.....	125,963	959,321	15,552	165,560	24,488	220,382	7,453 <sup>c</sup>	289,323 <sup>c</sup>	173,456 <sup>c</sup>	1,635,591 <sup>c</sup>

\* Minerals Yearbook, 1936.

<sup>b</sup> Average.

<sup>c</sup> Includes "clays or earths, artificially activated with acid or other material," as follows: 1930 (June 18 to Dec. 31), 2,663 short tons valued at \$100,779; 1931, 4,912 tons, \$184,381; 1932, 7,328 tons, \$267,560; 1933, 5,640 tons, \$253,291; 1934, 3,367 tons, \$232,664; 1935, 3,589 tons, \$212,036; not separately classified prior to change in tariff.

the vulcanizing batch, but the opinion is expressed that this trend cannot continue indefinitely. In the paper industry the increased use of domestic clay is said to represent progressive displacement of foreign clays, brought about in part by better preparation of the native product. On the other hand, clay has to compete in the paper industry with other mineral products such as talc, pyrophyllite, gypsum, lime carbonate, et cetera.

*Imports and Exports.*—Table 13 shows the quantity and value of clay imported for consumption in the United States from 1909 through 1934. The classification there adopted is far from satisfactory, and yet it has been used for a number of years. The figures in the total column show

a marked drop in tonnage and value since 1930, most of which appears to be due to the falling off of kaolin imports. This, as explained elsewhere, is accounted for largely by replacement of English by American clay. The exports of domestic clays are given in Table 14.

TABLE 14.—*Domestic Clay Exported from the United States*<sup>a</sup>

Year	Fire Clay		All Other		Total	
	Short Tons	Value	Short Tons	Value	Short Tons	Value
1925-29 <sup>b</sup>	55,316	\$434,842	54,028	\$ 782,927	109,344	\$1,217,769
1930	62,660	519,788	73,870	1,108,586	136,530	1,628,374
1931	45,314	329,112	61,389	915,743	106,703	1,244,855
1932	22,086	228,073	59,273	826,550	81,359	1,054,623
1933	32,432	264,595	66,093	970,293	98,525	1,234,888
1934	36,053	308,424	84,264	1,147,555	120,317	1,455,979
1935	49,949	380,604	101,524	1,484,465	151,473	1,865 069

<sup>a</sup> Minerals Yearbook, 1936. Statistics of exports of clays were not separately recorded before 1916. High unit value indicates processed clays, mainly filtering and decolorizing clays or fuller's earth, mainly from Pacific ports.

<sup>b</sup> Average.

#### PROSPECTING AND MINING

A knowledge of the geological characteristics of clay deposits is of considerable aid in prospecting for clays and in making a field examination. All natural exposures as well as artificial cuts should be carefully examined, not losing sight of the fact, however, that on slopes or steep surfaces there may be considerable weathering and sliding of the material, which covers up a portion of the material in place.

In unconsolidated deposits, a carpenter's auger attached to a sufficiently strong pipe may be used for boring the deposit. Such an outfit can be used to depths of 40 or 50 ft., but the system should be made in short sections, which can be connected by means of coupling. An auger outfit can also be used for horizontal boring. The data obtained from properly located boreholes can be used to determine both vertical and horizontal variations in the deposit. Where the ground is too hard to use augers, core drills may be employed.

If large samples are desired, test pits can be sunk in the deposit. Clay deposits are sometimes prospected by tunnels and shafts.

*Sampling.*—The Committee on Standards of the American Ceramic Society<sup>3</sup> recommends that for preliminary testing the body of clay shall be stripped of loose or foreign materials, and a series of straight, parallel trenches cut entirely across the outcrop. If the deposit is stratified and the beds dip, the trenches shall be cut across the dip. Each trench should be 12 in. wide and deep enough to yield 100 lb. of material. If natural outcrops are not available, preliminary trials may be made with a hand

auger, and test pits dug for further sampling. Should the deposit show beds that are visibly different from each other, they should be sampled separately. If preliminary tests show the deposit to be satisfactory, the deposit as a whole can be bored or drilled throughout its extent, spacing the holes not more than 100 ft. apart.

The samples collected from the different trenches are to be reduced to lumps not over 2 in. in diameter, mixed together, and reduced by quartering to 100 lb.

Too much stress cannot be laid on the fact that all samples collected should represent the average of the deposit or the bed. Furthermore, the clay should be carefully tested before a plant is erected. The writer has known of several plants erected on the site of improperly tested deposits; the result being failure of the enterprise.

Most clay deposits are worked as open pits, the method of excavation depending on the character of the clay, thickness and extent of beds, and character of the overburden. Where the deposit is small, or consists of beds of different quality, which it is desirable to separate, the clay may be dug by spades or mattocks. Selective mining of different beds is practiced in New Jersey, western Kentucky and Tennessee, and also in parts of California. Linton<sup>38</sup> states that in some California deposits over 20 ft. thick, beds as thin as 2 ft. may be separated in selective mining. With thicker beds, and a working face of not more than 20 ft. high, a steam shovel may be employed.

At some clay pits the face of clay, if not too high, may be undercut at the base, and wedges driven in at the top, thus causing a slice of the bank to fall and break up the clay, so that it can be more readily handled. If the clay deposit has a horizontal surface of some extent, it may be first loosened by plows, and the clay then gathered up by wheel or drag scrapers.

Shale banks are often blasted and the material that accumulates at the bottom gathered up by steam shovels. Occasionally shale is worked by a planer. This consists of a steel structure<sup>35,43</sup> carrying a power-driven steel chain provided with teeth, which scrape off pieces of shale. These pieces fall to the base of the face, and from there are carried by an endless belt to hoppers or vehicles. Such planers can cut a vertical or steeply sloping face. Depending on their construction, they can work on a straight line or cut a circular swath through an angle of 180°. A similar machine provided with small buckets has been used in soft clay.

For removal of overburden the methods used may be similar to those employed for excavating clay. In some places drag scrapers on cables are employed, or if the overburden is of sandy or gravelly character it can be removed by hydraulicking. At one shale bank near Seattle, Wash., where the stripping was 50 to 75 ft. thick, this method was used. The sale of the sand and gravel for concrete, etc., paid for the cost of stripping. The amount of overburden that can be economically stripped

depends on the thickness of the clay and its market value. Linton,<sup>38</sup> describing clay mining in California, states that the ratio of overburden thickness to that of clay varies from 1:1.2 to 1:1.75. In Tennessee as much as 35 ft. of overburden has been removed to dig 10 ft. of ball clay.

A unique type of mining is that employed in some of the North Carolina kaolin deposits. Circular pits 15 to 25 ft. in diameter, lined with cribbing, are sunk to the bottom of the deposit. The cribbing is fastened every few feet. When the clay is bottomed the cribbing is removed from the bottom up, and the pit is filled with stripping material. A more recent practice consists in using the large circular cribbed shafts as sumps into which the surrounding clay is washed by hydraulic jets. Bucket elevators raise the crude clay from the bottoms of the shafts, and discharge it into troughs leading to the washing plant. Hydrauliclicking is also used in the Cornwall district, England, where the pits may be 200 ft. deep or more.

In Florida the white clays have been excavated with a clamshell dredge floating in the pit. The material is dumped into barges or pumped ashore through large pipes to the washing plant.

Underground mining is commonly used only for extracting the better grades of bedded clay, and usually is carried out by the room-and-pillar method.<sup>27</sup> It has been extensively used in some of the fire-clay deposits in the coal measures of the Central and Appalachian States.

In Colorado, along the foothills, where the clay beds dip steeply, and are interbedded with sandstones, the beds are worked down the dip and along the strike, leaving pillars of the hard clay; timbers are also placed to hold the hanging wall.

The clay is hauled by different methods from the bank or mine to the stripping bins or manufacturing plant. At small pits, close to the plant, horse-drawn carts or small dump cars drawn by horse are often employed. For longer hauls, trains of cars hauled by electricity or steam are preferred. Where the mine opening is on a mountainside at some elevation above the valley, gravity planes or wire-rope trams may be used to bring the material down to the storage bins or plant.

Relatively few figures of cost have been published, and these are not always up to date. The following figures given by Tyler<sup>36</sup> were collected from several different sources:

In California the total costs for open-pit work ordinarily range between 20¢ and \$1.50 per ton. For mining alone the approximate figures are 10 to 20¢ a ton for scraper or power-shovel loading (up to \$1 if there is much hand sorting). Trimming out of the pit or carriage to a near-by bin costs 5 to 25¢ a ton, and incidental charges also range between 5 and 25¢ a ton. In eastern Canada, with wages at 27 to 40 or 50¢ an hour, total costs were 15.1 to 39.7¢ a ton for shale. Underground mining costs usually average higher, but they must be kept down to \$1 or less for mining, loading and sorting. The determining factor in every case is the cost per ton f.o.b. cars, which is only \$1 for certain cheap grades but ranges upward to \$5.50 for rarer varieties.



McMahon,<sup>43</sup> after making a careful study of clay-mining costs in the provinces of Ontario and Quebec, points out the difficulties in the way of obtaining accurate data.

#### PREPARATION, TESTS AND SPECIFICATIONS

*Preparation.*—Clays used for the commoner grades of clay products and even for fire brick do not undergo much preparation, except the removal of pieces of stone. Even ball clays are not put through any purification before shipment.

Kaolins, because of their sandy character when mined, are commonly washed.<sup>25</sup> At the usual type of washing plant the crude kaolin, if not already disintegrated with water, may be dumped into a log washer, which breaks it up. From this the water and clay pass into a sand box, where the coarse sand settles and is continuously removed by a sand wheel. The water, with suspended clay, mica, and fine to medium sand, passes next into a series of troughs, which have a pitch of about 1 in. in 20 ft., and a total length of 500, or sometimes even 1000 ft. As the water flows through this troughing the sand and much of the mica settle, so that at the end little but clay is left in suspension. It then runs through a 100-mesh screen to the settling tanks. At some plants an alkaline salt like sodium hydroxide is added to the water to help to deflocculate the clay, a practice that may be particularly desirable if the clay contains much fine silt.

When the clay has settled in the tanks, the clear water is drawn off and the creamy mixture from the bottom of the vats is pumped out and into the filter presses. The filter cakes, containing about 25 per cent water, are dried by artificial or natural heat, then broken up.

Dorr classifiers have been used at some plants for washing the kaolin.<sup>61</sup> Air separation is used sometimes, and it is probable that it will find greater favor in the future.<sup>86</sup>

Weathering may improve the plasticity, and soluble salts may be brought to the surface and washed away; on the other hand, new ones may form, such as ferrous sulphate from pyrite in the clay.

Other forms of preparation are carried out at the factory after the clay has entered the manufacturing process, and need not be considered here.

*Tests.*—Tests of clay are made primarily to measure their physical properties, and certain tests have been recommended by the Committee on Standards of the American Ceramic Society,<sup>3</sup> which apply to clay to be used in its fired condition.

*Specifications.*—While no standard specifications have been recommended, it is always possible for the consumer to prepare them himself, indicating what properties the raw material shall possess.

*Plasticity.*—There is no standard test for determining the plasticity of a clay, although several different ones have been suggested.<sup>46, 57</sup> It is often determined by feel.

*Fineness.*—The fineness of clay may be tested by washing the sample through sieves and determining the weight remaining on each. Sieves are not fine enough to separate the smaller particles of clay, and for this purpose the hydrometer method, as used successfully by the U. S. Bureau of Public Roads, can be employed. Similar results are obtainable by the pipette method. The ordinary elutriator is not to be recommended, even though it has been much used in Europe.

*Water of Plasticity.*—This is determined by weighing the freshly molded test piece, air-drying, then again weighing after oven-drying at 110° C. The difference in weight is water of plasticity, which is calculated in percentage of the dry weight.

*Shrinkage.*—The drying volume shrinkage is determined by placing a molded test piece in kerosene in a volumeter, of which there are several kinds.<sup>46,57</sup> After drying, the piece is soaked in kerosene for 12 hr., then placed in the volumeter to determine its dry volume. The percentage of shrinkage can then be determined in terms of the dry volume.

The linear shrinkage can be calculated from the percentage of volume shrinkage by the formula

$$\text{Linear shrinkage} = \left[ 1 - \sqrt[3]{1 - \frac{b}{100}} \right] \times 100$$

in which  $b$  equals percentage of volume shrinkage. The linear shrinkage can also be determined by making a line of known length on the freshly molded test piece and measuring again when dried.

For shrinkage determinations of the fired pieces, the same methods are employed using water instead of kerosene in the volumeter.

*Shrinkage and Pore Water.*—The data obtained in determining volume shrinkage are employed here. Thus, for shrinkage water:

$$t_1 = \frac{V_p - V_d}{W_d} \times 100$$

in which  $t_1$  = percentage of shrinkage water.

$V_p$  = plastic volume, c.c.

$V_d$  = dry volume, c.c.

$W_d$  = dry (100° C.) weight, grams.

Pore water is obtained from the formula

$$t_2 = T - t_1$$

in which  $t_2$  = percentage pore water.

$T$  = percentage of water of plasticity.

$t_1$  = percentage shrinkage water.

*Slaking Test.*—A 1-in. cube consisting of a mixture of 50 per cent clay; ground to pass a 30-mesh sieve, and 50 per cent potter's flint, is cut from a slab of the plastic material. This is dried in the room, then at 64° to

76° C., and finally at 110° C. It is cooled in a dessicator and then immersed in water on a  $2\frac{1}{2}$ -in. sieve. The time for it to disintegrate completely is noted.

*Transverse Strength.*—The plastic clay is molded into bars 7 in. long and 1 in. square. These are first carefully dried at room temperature, then at 64° to 76° and then at 110° C. They are cooled in a dessicator and broken in a machine in which they rest on supports 5 in. apart, the load being applied at the rate of about 100 lb. per minute. The modulus of rupture is calculated by the formula

$$MR = \frac{3Wl}{2bh^2}$$

when  $MR$  = modulus of rupture, lb. per sq. in.

$W$  = breaking load, lb.

$l$  = distance between knife-edges, in.

$b$  = breadth of bar, in.

$h$  = height of bar, in.

Since the clay shrinks in drying, the breadth and height of the bar must be measured before testing. Ten bars should be broken, and vary by not more than 15 per cent from the average.

*Firing Tests.*—Test pieces are fired over a range of temperature that depends on the type of clay, at a rate of 45° C. per hour up to a little below the point of drawing trials, and after that at a rate of 20° C. per hour. The fired trials are examined for color, hardness, shrinkage, porosity, etc. The fusibility is determined by grinding the clay to pass a No. 60 sieve, molding it into tetrahedra 30 mm. high and 7 mm. on a side at the base, and testing these in comparison with standard cones of similar size in a furnace with neutral or oxidizing atmosphere.

*Specific Gravity.*—The true specific gravity is determined in the usual manner in a pycnometer. The apparent specific gravity of a fired clay is determined by the formula

$$Ga = \frac{Wf \rightarrow}{Vf - (Sf - Wf)}$$

and the bulk specific gravity with

$$Gb = \frac{Wf}{Vf}$$

in which  $Wf$  = weight of fired piece, grams

$Vf$  = volume of fired piece, c.c.

$Sf$  = weight of saturated fired piece, grams.

*Absorption.*—For fired pieces this is determined by weighing the dry piece, soaking in water for 24 hr. then weighing again. The increase in weight is calculated in percentage terms of the dry weight.

*Apparent Porosity.*—This is calculated by means of the following formula

$$\text{Apparent porosity} = \frac{Sf - Wf}{Vf} \times 100$$

Saturation is obtained by soaking the test piece in distilled water at 20° C. for 100 hr., and boiling for 1 hr. during the first, twenty-fifth, forty-ninth and seventy-third hours.

#### MARKETING AND USES

*Marketing.*—Producers of the cheaper types of clay products, such as building brick, drain tile and paving brick, or even common red earthenware, with hardly any exception, obtain their clay from their own deposits. Only the kaolins, slip clays, and many refractory clays, as a rule, are mined by separate companies or individuals and sold to the consumers by the ton.

Some clays and shales are sold on a royalty basis, this being quoted<sup>86</sup> as 5¢ a ton for common clays and shales, 10¢ for moderate-duty fire clay, and 10 to 25¢ for high-grade fire clays. The white-burning clays bring the highest prices on the market, but for any one type of clay the price may vary, depending on the amount of preparation it goes through before being shipped.

Refractory clays are rarely shipped long distances to market, unless by water, but china clays, because of their relative scarcity, will stand a longer haul. The two great whiteware-potting centers of the United States are Trenton, N. J., and East Liverpool, Ohio, but the nearest important domestic sources of white clay are western North Carolina, the Georgia-South Carolina belt, and Florida. The chief markets for these

TABLE 15.—*Prices of Various Clays in the United States, 1931–1934<sup>a</sup>*

	1931	1932	1933	1934	
				January	December
China clay, f.o.b. mines South Carolina and Georgia:					
Crude lump No. 1. . . .	\$ 4.50–\$ 5 00	\$ 3 50–\$ 6 00	\$ 3 50–\$ 4 00	b	b
Crushed . . . . .	6.00– 8 00	4 50– 6 90	4 50– 5 00	\$ 5 00 <sup>c</sup>	\$ 6 00 <sup>c</sup>
Air-floated					
No. 1. . . . .	9 00– 15 00	6 50– 15 00	6 50– 10 00	b	b
No. 2. . . . .	5.50– 8 00	5 50– 6 00	5 50– 8.00	b	b
Florida, washed, crushed:					
Superwhite . . . .	12 50	11 75– 12 50	11 75– 12.75	12 75	12.75
Superplastic . . .	12 00	11 25– 12 00	11 75– 12 75	12 75	12 75
Delaware, No. 1. . .	14.50– 15 00	13 00– 14 50	14 00	14 00	14 00
English, f.o.b. United States port, lump, in bulk. . . .	15 00– 25.00	12 00– 21 00	14 00– 18 00	\$16 50– 25.00	\$15 00– 22.00

<sup>a</sup> Metal and Mineral Markets quotations, from Minerals Yearbook, 1934, U. S. Bureau of Mines.

<sup>b</sup> Not quoted.

<sup>c</sup> Water-washed, \$1.50 per ton extra; in paper bags, \$2.50 extra.

clays are in the Central and Northeastern States, and this means an appreciable freight rate, the latter from Georgia being given as \$5.80 to Trenton and \$8 or \$9 to New England points.<sup>85</sup> English clays may have to pay a freight rate of about \$3.50 a ton, and duty of \$1.50 a ton, and American china clays have to meet this competition.

Table 15 gives the prices for domestic kaolins prepared in different ways, and Table 16 the average value per short ton of various kinds of clay over a period of years.

TABLE 16.—*Average Values per Short Ton of Various Kinds of Clay Sold by Producers in the United States<sup>a</sup>*

Year	Kaolin and Paper Clay		Ball Clay	Slip Clay	Fire Clay	Stoneware Clay	All Kinds of Clay
	United States	South Carolina					
1909-13 <sup>b</sup> . . .	\$5 34	\$3.88	\$3.65	\$1.81	\$1.39	\$1.00	\$1 57
1925-29 <sup>b</sup> . . .	8 45	8.93	7.67	5.41	2.76	2.12	3 44
1930 . . .	7 29	7.61	7 91	6 02	2.38	1.93	3 16
1931. . .	6.65	6.84	7 71	7.10	2 54	2.30	3 32
1932 . . . .	5.83	6.40	6.57	9 72	2 83	1.66	3 74
1933 . . . . .	5.75	5 99	6.21	7.28	2 77	2 11	3.72
1934. . . . .	6.33	7 26	6.73	7.00	2.90	1.77	3.75
1935. . . . .	7.19	7.57	6.72	5 99	2.54	1.98	3 68

<sup>a</sup> Minerals Yearbook, 1936.

<sup>b</sup> Average.

*Uses.*—Clays may be used in their unfired or fired condition. The properties that govern the use of clays in their raw condition are color, texture, bonding strength and adsorptive qualities. The uses to which unfired clays are put include the following:

Paper clays, usually kaolins of residual or sedimentary character. Whiteness, fineness and uniformity of texture are of prime importance. Somewhat similar clays are used as fillers in fabrics, and in ultramarine manufacture. This group may be designated as "fillers." The requisite properties of filler clays as specified by different consumers, either as groups or individuals, are somewhat conflicting, but whiteness and freedom from grit seem to be the essentials.<sup>89</sup>

Bleaching clays, employed in the decolorizing of mineral and organic oils, include some of the clays known as bentonites (see chapters VI and VII).

Bonding clays, which have strong bonding qualities, are widely used for binding together the sand grains in synthetic molding sands. Such clays are commonly tested when mixed with sand, the properties of strength and permeability being important. They are commonly fire clays, although bentonite is also much used for this purpose. Under this

head might also be included the clay used for making stabilized roads, in which is employed a properly proportioned mixture of gravel or crushed stone, sand and clay. The clay should be tested for its cohesiveness, and this is commonly done by the Atterberg test.<sup>57</sup>

In its fired condition, clay may be used for a variety of purposes, prominent among which are structural, refractory, decorative and household. The deciding factor is the physical behavior of the clay in both its raw and its fired condition. While different types of clay may be designated by certain names, such as china, brick, stoneware, terra cotta, etc., such terms refer only in a general way to their properties, and do not indicate restricted uses. Thus a clay suitable for brick might also be used for drain tile, or a paving-brick clay could also be used in sewer-pipe manufacture. Furthermore, many clay products other than the cheapest are often made of a mixture of clays, in order to get a material with suitable properties. Architectural terra cotta and chemical stoneware are usually made from a mixture of clays. Whiteware contains kaolin, ball clay, ground quartz and ground feldspar. Any reference to the uses of clay, therefore, might better be expressed in terms of physical properties than in terms of names. The following classification, given by Parmelee,<sup>47</sup> brings out these points.

### *Use Classification of Clays*

- I. Clays burning white or cream, not calcareous.
  - A. Open-burning clays (i.e., still distinctly porous) at cone 15 (2606° F.).
 

Uses: If of good color or of good strength, is used for pottery. If of good or high degree of refractoriness, used for various refractories; if also of good color, used for special refractories (e.g., pots for melting optical glass).

    1. Low strength. Type: residual kaolins of North Carolina.
    2. Medium and high strength. Type: secondary kaolins of Florida and Georgia.
  - B. Dense burning (i.e., become nearly or completely nonporous) between cones 10 and 15 (2426° and 2606° F.). Medium to high strength, medium shrinkage.
    - a. Nonrefractory clays.
      3. Good color. Uses: ~~pottery~~, including certain whiteware, porcelains, stoneware.
      4. Poor color. Uses: stoneware, terra cotta, abrasive wheels, zinc retorts, face brick, saggars.
    - b. Refractory clays.
      5. Good color. Uses: refractories, especially for glass if they do not overburn seriously for five cones (about 1800° F.) higher. Also uses stated in 3.
  - C. Dense burning between cones 5 and 10 (2426° to 2606° F.) and do not overburn seriously at five cones (about 1800° F.) higher than the temperature at which minimum porosity is reached.
    - a. Nonrefractory clays, medium to high strength, medium shrinkage.
      6. Good color; usually reach minimum porosity between cones 5 and 8 (2246° to 2354° F.). Type: ball clays. Uses: pottery, whiteware, porcelain and stoneware.
      7. Poor color. Uses: stoneware, terra cotta, abrasive wheels, zinc retorts, face brick, saggars.

b. Refractory clays.

8. Dense burning at cone 5 (2246° F.); do not seriously overburn for 12 cones (about 432° F.) higher; highly refractory; softening point at cone 31 (3,182° F.) or higher; bonding strength minimum, 325 lb. per sq. in. Use: graphite crucibles for melting brass.
9. Dense burning at cone 8 (2354° F.); not overfiring at cones 13 or 14 (2550° F.). Strength and softening point as in 8. Use: steel crucibles.
10. Dense burning at cone 8 (2354° F.); not overfiring at cone 15 (2606° F.). Bonding strength, 250 lb. per sq. in. or higher. Softening point, cone 29 or higher. Use: glass pots.

II. Buff-burning clay.

A. Refractory clays.

- a. Open burning (5 per cent porosity or more) at cone 15 (2606° F.) or above. Indurated. Nonplastic or slightly plastic (unless weathered). Type: flint clays.
  11. Alumina 40 per cent or less. Use: refractories.
  12. High alumina (over 40 per cent). Type: diaspore clays. Uses: refractories, abrasives.
- b. Open burning (5 per cent porosity or more) at cone 15 (2606° F.) but plastic.
  13. Silica 65 per cent or less. Uses: firebrick and other refractories, terra cotta, sanitary ware, glazed and enameled brick.
  14. High silica (over 65 per cent). Type: many New Jersey fireclays. Uses: firebrick and other refractories.
- c. Dense burning (porosity under 5 per cent) between cones 10 and 15 (2426° to 2606° F.).
  15. Medium to high strength, not overburning for five cones (about 1800° F.) higher than point of minimum porosity. Uses: glass pots, firebrick, saggers, and other refractories; architectural terra cotta, sanitary ware, enameled and face brick.
- d. Dense burning (porosity under 5 per cent) at cone 10 (2426° F.) or lower.
  16. (See 8.) Uses: zinc retorts, firebrick, saggers and miscellaneous refractories.
  17. (See 9.) Uses: architectural terra cotta, sanitary ware, enameled and face bricks.
  18. (See 10.) face bricks.

B. Nonrefractory clays.

- a. Open burning (5 per cent porosity or more) at cones lower than 10 (2426° F.).
  19. High or medium strength. Uses: architectural terra cotta, stoneware, yellow ware, face brick, sanitary ware.
  20. Low strength. Use: brick.
- b. Dense burning (porosity under 5 per cent) at cones lower than 10 (2426° F.).
  21. High or medium strength. Uses: architectural terra cotta, stoneware, abrasive wheels, sanitary ware, face brick, paving brick.

III. Clays burning red, brown, or other dark colors.

- A. Open burning (do not attain low porosity at any temperature short of actual fusion).
  22. Medium or high strength. Uses: brick, drain tile, hollow blocks, flower pots, pencil clays, ballast.
  23. Low strength. Use: brick.
- B. Dense-burning clays.
  - a. Having a long vitrification range (5 cones or about 1800° F.).
    24. High or medium strength. Uses: conduits, sewer pipe, paving brick, floor tiles or quarries, electrical porcelain, cooking ware, silo block, artware, face brick, architectural terra cotta, roofing tile.

25. Low strength. Uses: as dust body in manufacture of electrical porcelain, floor tile, building brick.
  - b. Having a short vitrification range:
    26. High or medium strength. Uses: building brick, face brick, hollow block, flower pots.
    - c. Highly fusible, forming a glass at about cone 5 (2246° F.)
  27. Slip clays.
- IV. Clays burning light gray or light cream.
28. Containing calcium or magnesium carbonate or both. Never attain low porosity. Very short heat range. Use: common brick.

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## CHAPTER XIII

### DIATOMITE

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**D**IATOMITE is a hydrous or opaline form of silica, commonly known as diatomaceous earth, diatomaceous silica, kieselguhr and, incorrectly, as infusorial earth. Obsolete locality or variety names such as bergmehl, desmid earth, white peat, randanite, polierschiefer, tellurine, tripolite and ceyssatite are no longer favored. Moler and Celite are trade names that have become associated respectively with Danish diatomites and those from Lompoc, Calif., and have been widely used in the technical literature.

#### COMPOSITION AND PROPERTIES

*Chemical Composition.*—Diatomite is essentially hydrous silica (2 to 10 per cent combined water), but is frequently associated with organic matter (from traces up to over 30 per cent) and with varying amounts of inorganic impurities such as sand, crystalline silica, clay, volcanic ash, calcium carbonate, magnesia, soluble salts. In the natural or crude state, diatomite contains from 10 to 65 per cent or more of free water. Table 1 shows the wide range in chemical composition of diatomite samples from various deposits.

*Physical Properties.*—Pure varieties have low density and are friable, porous, chalklike masses, either stratified or massive; in color, usually white or light shades of gray, buff or green. Varieties containing organic matter range from oozes in lake bottoms to peatlike materials; impure varieties may be more or less claylike, sandy, calcareous or flinty.

Color: white, cream, gray, tan, brown, greenish to nearly black. Hardness: pure varieties easily friable and apparently soft 1 to 1.5 because of porosity, actual hardness of the microscopic particles 4.0 to 6.0. Specific gravity: 1.9 to 2.35. Apparent density (dry block form): 0.40 to 0.60 (25 to 37.5 lb. per cu. ft.). Apparent density (dry powder): 0.08 to 0.25 (5 to 16 lb. per cu. ft.). Crystal system: previously considered amorphous, but there is some evidence of faint crystallinity similar to high cristobalite.<sup>19, 24, 30, 39, 45</sup> Melting point: 1400° to 1750° C. Streak: white. Luster: dull to earthy. Cleavage: none. Fracture: parallel

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<sup>19</sup> References are at the end of the chapter.

with bedding planes, conchoidal or irregular. Transparency: opaque. Tenacity: crumbly to brittle. Solubility: insoluble in acids (except hydrofluoric), soluble in strong alkalis. Absorption: 1.5 to 3.5 times its weight of water. Index of refraction: 1.42 to 1.48. Thermal conductivity, block form (Celite): perpendicular to strata 0.79 B.t.u. per sq. ft. per hr. per in. thickness per 1° F. at 1000° F. mean; parallel to strata 1.13 B.t.u. per sq. ft. per hr. per in. thickness per 1° F. at 1000° F. mean. Thermal conductivity, powdered form (Sil-O-Cel, 16.0 lb. per cu. ft. density): 0.37 B.t.u. per sq. ft. per hr. per in. thickness per 1° F. at 86° F. mean; 0.73 B.t.u. per sq. ft. per hr. per in. thickness per 1° F. at 1000° F. mean.

TABLE 1.—*Chemical Analyses of Diatomaceous Earths from Different Localities, Moisture-free Basis*

Constituent	Lompoc, Calif.	Tlax- cala, Mexico	Mont- erey Co., Calif.	Los Angeles Co., Calif.	Mary- land	Vir- ginia	Ger- many	Algeria
Silica (SiO <sub>2</sub> )	89 70	80 31	83 20	75.62	79 55	76 10	68 30	71.99
Alumina (Al <sub>2</sub> O <sub>3</sub> )	3.72	9 73	5.15	5 75	8 18	9 13	1 57	3 00
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	1 09	2 80	1.58	2 56	2 62	2.56	2.37	1 12
Titanium oxide (TiO <sub>2</sub> )	0.10	0.74	0 20	0 20	0 70	0.56	0.11	0.13
Manganese oxide (Mn <sub>2</sub> O <sub>3</sub> )	0.02			0 05			0 09	
Lime (CaO)	0.35	0 18	1 30	3 30	0 25	1 85	Trace	8.15
Magnesia (MgO)	0.65	0.53	1 41	1 50	1 30	1.65	0.18	2 30
Alkalies (as Na <sub>2</sub> O)	0.82	1.44	1.40	2 16	1.31	1 53	0.84	0.63
Ignition loss (combined water, CO <sub>2</sub> and organic matter).	3.70	4.48	5.95	8.22	5 80	6.68	26.50	12 68
Total.. . . .	100 15	100.21	100.19	99 36	99.71	100 06	99.96	100.00

*Microscopic Appearance.*—All diatomites consist of the siliceous remains of diatoms, in whole or partly fractured condition (sometimes associated with minor amounts of sponge residues, radiolaria, silico-flagellae, and other microscopic organisms, both plant and animal). There is an infinite variety in the types and conditions of diatom residues found, there having been identified some 8000 different species. The shape, size and structure of these vary enormously (Figs. 1 and 2).

#### ORIGIN AND MODE OF OCCURRENCE

Diatomite is of organic origin, consisting of the fossil remains of microscopic aquatic plants known as diatoms, of the order Bacillaria. These minute organisms thrive everywhere under suitable conditions of light, moisture and food supply. Each tiny plant is enclosed in a siliceous envelope, and when the organism dies it sinks to the bottom of the water, where the organic matter is more or less decomposed. The siliceous cases persist because of their imperishable nature, and thus form a deposit.

Diatoms grew in great profusion during various geological periods, particularly the Miocene, and the most important economic workings today are in the fossil deposits of this period. Some, however, are recent and others are in the course of formation now. Since diatoms live in fresh, marine and brackish waters, deposits of diatomite are likewise of corresponding origin. No large economic deposits are of earlier age



FIG. 1.—PROCESSED DIATOMACEOUS FILTER AID, JOHNS-MANVILLE CORPORATION.  $\times 200$ .  
FIG. 2.—FLORIDA DIATOMACEOUS EARTH (CALCINED).  $\times 200$ .

than the Tertiary. In the United States, there are deposits of both marine and fresh-water origin; in geologic age they range from the Cretaceous to the Recent.

The deposits near Lompoc, Calif., are of plankton marine origin and belong to the Monterey series of the upper Miocene. The main deposit today consists of 1400 ft. or more of stratified diatomite, now gently folded into a series of synclines and anticlines. The area of the Celite deposit is some 4000 acres. Deposits in Los Angeles and Monterey

Counties, California, are of different degrees of purity and of limited size. These are likewise marine Tertiary deposits, as are some undeveloped deposits in lower California.

There are a considerable number of fresh-water deposits in the Western States. All the known deposits of Oregon and Washington were laid down in fresh water in late Tertiary or Pleistocene times. They exist mainly as horizontal beds formed in prehistoric lakes. The numerous deposits of Nevada and some others of the Western States (Arizona, Utah, Idaho, New Mexico) also are of fresh-water origin. They occur as stratified basin-shaped beds, overlain and interlain with sedimentary and igneous formations.

Marine Tertiary deposits occur in Maryland and Virginia. The beds are intermixed and interstratified with sand and clay along the Potomac and Rappahanock Rivers and their tributaries. In New York, New Hampshire, the maritime provinces of Canada and Florida, there are recent deposits in ponds, peat bogs, marshes and small lakes.

The Algerian deposits are marine Miocene and occur in formations somewhat similar to those of the California coast regions. The French deposits are principally of fresh-water origin, having accumulated in Tertiary and Quaternary lake beds. Deposits in the Lausitz district, Germany, and those near Bilin, Czechoslovakia, are in an area of Tertiary basalt. The Lüneburger Heide deposits were formed in the last interglacial period and lie in water-soaked beds covered with soil and sand. The Danish deposits (late Tertiary) are of great thickness, intimately mixed with clay. They are often exposed in sea cliffs. Japanese deposits (Tertiary and Pleistocene) are small and basinlike.

The source of silica for the great diatomaceous deposits has been considered by numerous geologists. Since diatoms are aquatic plants, the silica to be utilized must be in solution or the organisms must have the capacity to extract silica from suspended silicates such as clay, etc. Plant physiologists investigating the role of silica in the lower plants are not in full agreement as to the manner by which the diatom obtains its silica and deposits it as the characteristically thin and highly ornamented membrane of opal. However, since nearly all natural waters contain some dissolved silica and it appears that diatoms have limited capacity at least to extract silica from suspended silicates, it is believed that these sources are sufficient to account for the growth of diatoms as found usually today and possibly also for some of the smaller diatomite deposits. It is doubtful, however, whether these sources could be adequate to explain the more extensive deposits, particularly of the Miocene period. It must be assumed, therefore, that when these great beds were laid down the waters contained relatively high concentrations of dissolved silica.

A relationship between volcanism and diatomaceous sediments was first advanced by Whitney,<sup>44</sup> who pointed out that silica in available form

could be supplied by volcanic agencies in the form of ashes, pumice, etc. Taliaferro<sup>40</sup> has elaborated this viewpoint and has associated the Miocene deposits with contemporary submarine volcanism.

#### DISTRIBUTION OF DEPOSITS

Deposits of diatomite of varying degrees of purity and size are found rather widely but not extensively throughout the world. The most important deposit is near Lompoc, northern Santa Barbara County, California.

*United States.*—Deposits are found in many states, as follows: (1) California: in addition to those at Lompoc, there are deposits in Los Angeles County, near Covina and Torrance; in Monterey County, near Bradley and Monterey and in Shasta County, Pit River district (undeveloped). Minor occurrences are in Orange, Fresno, San Luis Obispo, Sonoma, Stanislaus and Lake Counties and on Santa Catalina Island; (2) Oregon: near Harper, Malheur County; Terrebonne, Deschutes County, and in Baker, Grant, Klamath, Wasco, Union, Crook, Lake and Wheeler Counties; (3) Washington: near Ellensburg, Kittitas County; in Grant County, Skagit County, and in Puget Sound districts; (4) Nevada: near Carlin, Elko County; Virginia City, Storey County; Lovelock, Humboldt County; Reno, Washoe County; Black Springs, Nye County; Lovelock, Churchill County; near Basalt, Esmeralda County, and in numerous other localities; (5) Arizona: in the San Pedro Valley, Graham County; near Mammoth, Pinal County and in the Bradshaw district, Yavapai County; (6) New Mexico: near Española, Santa Fe County and Rio Arriba County, and near Socorro, Socorro County; (7) Utah: in Utah, Box Elder and Beaver Counties; (8) Idaho: in Blaine, Owyhee and Washington Counties; (9) South Dakota: near Hermosa, Custer County; (10) Maryland: at Herring Bay, on the west side of Chesapeake Bay, Popes Creek, along the Patuxent River near Dunkirk and along Lyons Creek; (11) Virginia: commercially unimportant deposits at Richmond, along the Rappahanock River, in King George County and northwest of Petersburg, Dinwiddie County; (12) Florida: near Eustis and Clermont, Lake County, in Polk County and east of Pensacola; (13) New York: several localities in Herkimer County, in Suffolk County and near Cold Spring Harbor, Long Island; (14) Connecticut: near Stepney, Fairfield County; (15) Massachusetts: near South Framingham, Middlesex County; (16) Maine: near Blue Hill and Surry, Hancock County, and South Beddington, Washington County; (17) New Hampshire: pond deposits near Canaan, Grafton County; Center Ossipee, Carroll County; Laconia, Belknap County, and in Cheshire and Coos Counties; (18) Vermont: Caledonia County; (19) New Jersey: near Drakesville, Sussex County and in Morris County; (20) South Carolina: Williamsburg County;



(21) other states: localities have been reported in Nebraska, Alabama, Colorado, Delaware, Georgia, Montana and the Pribilof Islands, Alaska.

*Canada.*—Diatomite is found in British Columbia along the Fraser and Quesnel Rivers, at Prospect Lake near Victoria, and at other localities; in Nova Scotia, in Colchester, Cumberland and Pictou Counties. The principal deposits at present are near New Annan and on Digby neck. There are also deposits in the southern portion of New Brunswick, in Montcalm and Port Neuf Counties of Quebec and in the Muskoka region of Ontario. The deposits of Canada have been described in detail by Eardley-Wilmot.<sup>13</sup>

*Mexico.*—The principal deposit is in the state of Tlaxcala; others in states of Jalisco, Michoacan, Colima (Revillagigedo Islands) and Tepic (Las Tres Marias Islands).

*South America.*—Peru, near Arequipa; Chile, in the Atacama desert; in Tacna, Valparaiso and Nuble provinces and on Chiloe island; Brazil, Bahia and Minas Geraes.

*Europe.*—There are deposits of diatomite in Denmark, principally on the islands of Mors and Fur, lying in the Lim Fjord, also at Hollerup and Fredericia; in Germany, in the Vogelsburg, Hesse, between Altenschlirf and Steinfurt and in Lausitz, near Bilin, on the borders of Saxony and Czechoslovakia. The principal works are at Seifhennersdorf. Deposits also occur in Klieken, near Coswig, in Anhalt. The most important deposits occur in the Lüneburger Heide district, Hannover, where there are some 20 to 30 working pits in this area that may be classified into four producing areas: (1) Unterluss, Oberohe, Neuohe, Wiechel; (2) Munster; (3) Luhetal, along the upper Luhe valley, Bispingen, Hutzel, Steinbeck, Grevenhof, Schwindebeck; (4) Suderburg, Hamerstorf and Hösseringen. In France the present most important deposits are near Auxillac, north of Murat, Joursac, and Celles, all in the Department of Cantal. Other deposits, some of past importance, are in the Departments of Puy-de-Dôme, Haute-Loire, Ardèche and Aveyron. The active Auxillac quarries are in the upper Miocene; the older deposits in the Auvergne are principally Quaternary. Diatomite also occurs in: (1) Austria near Limberg; (2) South Bohemia, near Forbes (Borovany) and numerous other deposits; (3) Finland, lake deposits near Lavitaipole and Kymmene; (4) Hungary, near Zastreba; (5) Soviet Union near Pjatigorsk in Caucasian Mountains and Kreise Akhalzykh (Tiflis); (6) Georgia, District of Akhaltzik, near Kissatib and Saram; (7) Italy, near Santa Fiora, Bagnolo, Castel del Piano, between Tambolina and Metauro and in Tuscany south of Florenz at Monte Amiata; (8) northern Ireland, along the Bann River, in Londonderry and Antrim Counties; (9) Spain, in Province of Abacete, near Elche de la Sierra, also in the provinces of Castille, Seville and Cordoba; (10) Portugal, in the district of Samtarem. Norway, Sweden, and Yugoslavia have minor diatomite occurrences.

*Africa.*—Algeria, the principal deposits are in the province of Oran. The more important localities are: Saint Lucien near Saint Denis du Sig, Cassaigne, Ouillis and Pont du Chelif. Shipments are from Oran and Mostaganem. Deposits occur also in Kenya, Madagascar, Egypt, Nigeria, Union of South Africa.

*Japan.*—Numerous localities have been reported on the Island of Hokkaido (provinces of Shirabeshi, Kitami, Teshio and the Oshima peninsula); others on the Island of Kiushiu, prefectures of Kagoshima and Kummamoto and also on the small islands of Oki.

*Netherlands Indies.*—Diatomite occurs near Cheribon, in Java, and in Sumatra.

*Australia.*—Deposits are in Queensland, New South Wales, Victoria, Western Australia and Tasmania. New Zealand also has some occurrences. None of these have assumed commercial importance, except partly meeting local requirements. The deposits of Australia and New Zealand have been described rather fully in the literature.<sup>11,18,31</sup>

#### POLITICAL AND COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

It has been characteristic of the diatomite business that it has been developed and most requirements supplied by large individual companies or by trade organizations. Thus, in the United States, the Celite Company and the Johns-Manville Corporation pioneered the industry and continue to supply by far the greater tonnage for domestic consumption and also considerable tonnages for export shipments, particularly for filtration and filler uses. The International Diatomite Industries, Ltd., operating principally Nova Scotian deposits, is at present the leading company producing in Canada. In Germany the Vereinigte Deutsche Kieselguhrwerke G.m.b.H., Hamburg, has largely controlled the production and marketing of kieselguhr in that country. It represents a trade organization correlating the production of numerous works, principally in the Lüneburger Heide district. In France is La Société de Silices Fossiles de France, at Paris, which controls much of the Algerian diatomite. In Denmark, Moler Products, Ltd. and Molerindustri A/S largely control the industry. In Soviet Russia production and shipping is controlled by the Government. Private companies operate in north Ireland, Spain, Australia, Italy, Japan, etc. In the United States and Canada many minor companies and organizations have operated different deposits at different times. While most of these have been of temporary existence, with many ownership changes, some of the smaller producers have operated for many years and have been successful in specialties.

A feature of the diatomite industry in the United States has been a rather high degree of technical proficiency, not only in product and process development but also in its ability to develop new uses and to supply

technical service to the consuming industries. In North America, and to some extent elsewhere, the trade has come to expect distinct uniformity of grade and high-quality standards in diatomaceous products, except where it is used locally or when the lower quality products are utilized in the cheaper fields of building materials, ceramic insulations and concrete. The present developed United States resources appear adequate to meet all requirements for many years to come.

The first modern use of diatomite to any extent was in central Europe, particularly Bohemia, where it was employed principally as a polishing medium and as a clarifying agent in pharmacy. Beginning about 1860, with the development of the German deposits, came uses as an absorbent for nitroglycerin, as a source of silica for water glass, as a constituent of heat-insulating compositions and as a raw material for building materials. Later began the development of the Algerian, French, Italian and Danish deposits. In the United States small amounts of diatomaceous earth in small fresh-water deposits in the Eastern States, New England, New York, New Jersey, Maryland and Virginia were mined and used as an abrasive, filler and absorbent as early as 1850. The real development of the American diatomite industry began with the exploitation and operation of the deposit at Lompoc, Calif., about 1900. Production was very low until 1915, when financial backing was provided and, with a progressive management, the industry really began to develop. The field of filtration and development of efficient filter aids was pioneered (particularly for sugar refining) and the field of insulation was greatly broadened. High-temperature industrial insulation may be stated to have begun with the application of diatomite for these purposes on a sound technical basis, and the requirements of government and industry during the war period aided the new industry materially. Production increased steadily and in some products rapidly, reaching a maximum in 1929. During the period of economic depression, production of diatomite fell off, particularly for insulations and building materials. In the direct consumption industries, such as sugar refining, filtration generally, and in many filler uses, the demand for diatomaceous products held up comparatively well in the United States and throughout the world.

Published production statistics for diatomite have been incomplete and somewhat confusing. Early figures are particularly unsatisfactory. Since the figures that have been compiled may be obtained from government publications and other books readily accessible,<sup>5,11,13,31</sup> they will not be duplicated here in further detail. Production in the United States since 1921, in three-year periods, is shown in Table 2. World production figures for diatomite are given in the Imperial Institute and United States Government publications and annually in *The Mineral Industry* (McGraw-Hill Book Co., New York). Today, the principal producing nations are the United States, Germany, Denmark, Soviet Republic,

Algeria, Japan (including Chosen), and France. Not unimportant quantities are produced in North Ireland, Spain, Australia, Canada, Italy and Hungary. Minor producers are Norway, Sweden, Chile, Netherlands Indies, Mexico, Portugal.

TABLE 2.—*Production of Diatomite in the United States*

Years	In Three-year Period, Short Tons	Average per Year, Short Tons
1921-1923	162,768	54,256 <sup>a</sup>
1924-1926	219,331	73,110 <sup>a</sup>
1927-1929	236,426	95,475 <sup>b</sup>
1930-1932	248,273	82,758 <sup>b</sup>
1933-1935	244,342	81,447 <sup>b</sup>

<sup>a</sup> From The Mineral Industry of the British Empire and Foreign Countries—Diatomaceous Earth. Imperial Institute. His Majesty's Printing Office, London, 1928.

<sup>b</sup> From Minerals Yearbook, 1935. U. S. Bur. Mines. Government Printing Office, Washington, 1935.

The greatest diatomite resources, undeveloped or in reserve, are in the United States, Denmark, the Soviet Republic, Japan and Chosen, Algeria, Germany, Kenya (unproved) and, to a minor extent, Canada. Of all these, the greatest quantity of good quality material is distinctly in the United States. Only totally unimportant quantities of the best quality filter and filler material have come from Denmark, Soviet Republic, Japan or Algeria. The chief importing nations at present are the United Kingdom (Australia, England, Ireland, India), Canada, France, Norway, Poland, Cuba, Peru, Italy, Yugoslavia, Argentina, Java, Japan. Important aspects of diatomite production since 1935 are as follows: (1) leading position maintained by United States; (2) some revival of German industry (although believed not to have equaled production prior to 1930); (3) decrease in Algerian production and export, owing to economic conditions in France and the value of the franc; (4) increase in production in Soviet Russia, including Georgia, in Japan and north Ireland.

The outlook for the immediate future is considered favorable. With more stable economic conditions, world production may be expected to exceed 200,000 tons annually, with United States production passing 100,000 tons. With further development of uses and lowering of prices, total production may be expected to increase steadily for some decades. There is, however, no immediate expectation of revolutionary advances that would boost production unusually rapidly.

#### PROSPECTING, EXPLORATION AND MINING

In the dry, compact, stratified deposits, samples may be obtained from the surface outcrops and examined microscopically, physically or chemically. The purer beds are thus determined and further prospecting

results. Diatomite lends itself to core drilling readily and in some places prospecting is done in this manner. In others, trenches, tunnels, and vertical shafts of narrow cross sections are used to examine the diatomite away from surface conditions of alteration and leaching. These are more satisfactory methods of examination because of the ease with which the diatomite can be excavated. The openings permit a careful examination of the appearance of the material, its stratification, compactness, and inclination of beds. In the wet bog, lake, or swamp deposits, auger drills are often used, so that one foot of material can be brought up at a time. For under-water sampling, a casing is driven into the mud and boring is done inside the pipe. Tonnages are based on the amount of dry material in a cubic foot of the earth.

Where the diatomite is compact, the usual practice in the United States is to quarry on the surface with gasoline, steam, or Diesel shovels, and dragline hoists. In some locations, underground methods are employed, using simple shrinkage or room-and-pillar stoping. In most western United States deposits, the diatomite requires little or no timbering if pillars are left to support the roof. At Lompoc, Calif., gasoline and Diesel shovels load diatomite in the quarries into large dump trucks, which haul to vertical storage shafts. A 9500-ft. tunnel is electrified for underground hauling to the milling plants.

In eastern Canada and northeastern United States the diatomite is recovered from bogs, lakes and swamps. Here the diatomite is mined by dredging or by preliminary draining. After draining of water, recovery is by hand shoveling into dump cars, by bucket dredges, or by suction dredges. The wet diatomite is piled up to drain off free water, then spread out to air-dry before treatment.

#### PREPARATION FOR MARKET, TESTS AND SPECIFICATIONS

The method of preparation for market depends on the mode of occurrence, composition, and uses intended. In Denmark the impure, clayey material is simply milled and used for fabrication of ceramic articles (industrial and building). When it occurs in recent deposits, as in peat bogs, ponds, etc., the material is dredged, classified, dried and calcined (as in New York State and Florida). When coming from pits, as in the Lüneberger Heide, the material is dug, mostly by hand, calcined in lump form (or in special furnaces), elutriated, classified, et cetera.

In the western United States deposits, the crude earth from open quarries, cuts, etc., is field dried to some extent and then transported for milling. The large producers, however, have equipment and processes for drying or for drying and milling simultaneously. Diatomite products may be classified broadly as brick or block, crushed aggregates and pulverulent powders. Diatomite brick, with clay or other binders and with or without organic filler to be burned out, are fabricated with

conventional equipment: blending bins, puggers, presses, driers, kilns, sizing equipment, etc. The principal manufacture of brick and pressed block is in the United States, England, Germany and Austria. Aggregates are prepared by crushing, screening, and grading—all with more or less conventional equipment.

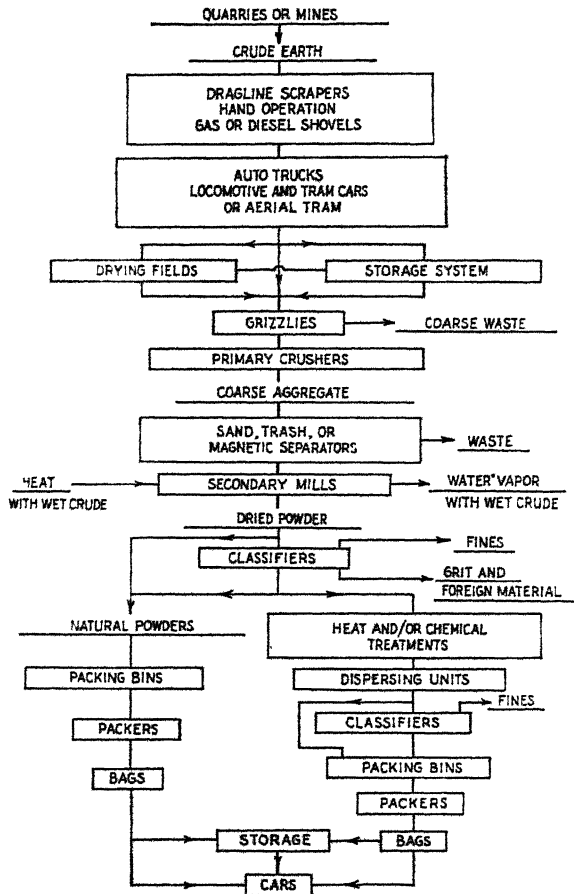


FIG. 3.—FLOWSHEET FOR DIATOMITES FROM DEPOSITS IN WESTERN UNITED STATES.

In the preparation of diatomaceous powders, the crude (field dried or wet) is conveyed to storage system or directly to trommel screens or grizzlies, then to primary crushers, mechanical traps for removal of rock, trash, flint or coarse impurities of any kind, thence to driers or to the secondary mills in combination with driers, thence to air classifiers for removal of finer grit and foreign matter and sometimes to classify pneumatically according to particle size. Many types of primary crushers may be employed. For secondary milling, mills on the swing-

hammer principle or centrifugal fans are preferred. Some products are calcined and chemically treated; the dried powders go to rotary or stationery kilns or to treatment tanks. Following either heat or chemical treatment, final dispersion of the powders is generally necessary and this is accomplished by special mills or blowers. In some instances, further purification and classification is effected pneumatically, subsequent to calcination. Water classification is carried out to some extent in Germany.

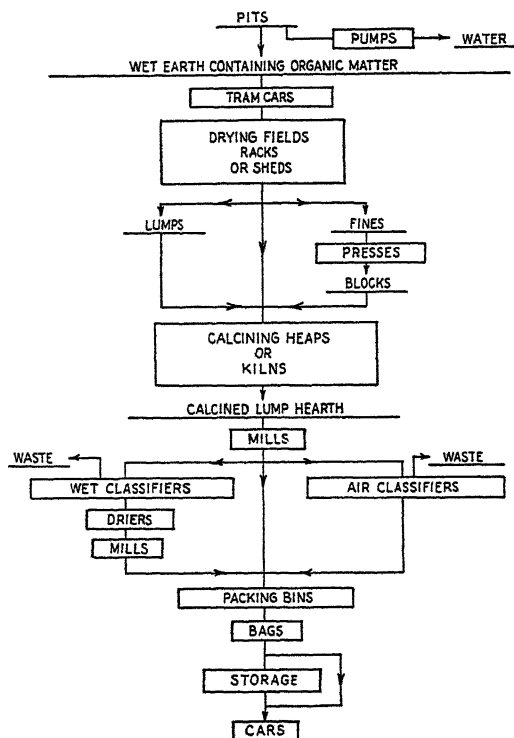


FIG. 4.—FLOWSHEET FOR DIATOMITE FROM PIT MINING, EUROPEAN PRACTICE.

Three idealized and simplified flowsheets are shown herewith for different methods of processing raw diatomite (Figs. 3 to 5).

*Tests and Specifications.*—Crude diatomite is evaluated on the following properties: freedom from impurities (sand, volcanic ash, crystalline silica, organic matter, clay, lime, soluble salts, etc.); microscopic structure (types of diatom forms and their condition, whole or broken, and relative proportions of different diatom forms, presence or absence of exceedingly fine particles); apparent or bulk density; friability (behavior on milling); color; opaline silica content.

The value of diatomaceous products is determined by: microscopic structure, bulk density (loose, tamped or on vibration); absorptive capac-

ity (water, oil or other liquids); chemical composition (iron, alumina, manganese, rare metals, alkalis, organic matter, etc.); specific gravity (care required for high accuracy); screen analysis (usually wet screening for finer than 30 mesh); particle size (generally by sedimentation methods); color (dry and in water, oils, etc.); conductivity thermal (standard methods); moisture (free); combined water (loss on ignition, in absence of organic matter and carbonate); special tests such as filtering efficiency and workability as admixture.

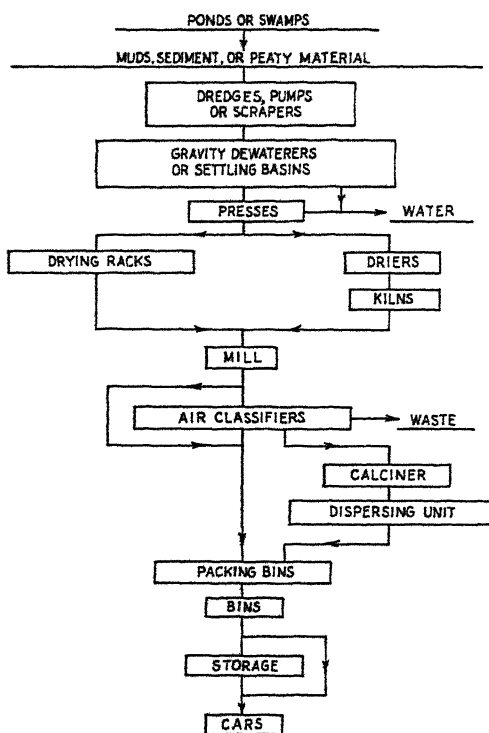


FIG. 5.—FLOW SHEET FOR POND OR BOG DEPOSITS, EASTERN UNITED STATES.

Specifications for diatomaceous products vary greatly because of the wide diversity of uses and requirements. For low-priced crude near sources of supply, the only requirements are bulk density and moisture content. For some specialized filtration and filler uses, definite chemical and particle-size specifications are required. For some polish and abrasive uses there are exacting requirements for mesh size and freedom from grit. For some filler uses there are equally stringent requirements for color and particle size. Many customers have set up special requirements that are not publicized. There are several government specifications, particularly for insulating materials, admixtures and fillers. The



principal producers have also supplied specifications for many of their standard products. It is frequent practice to submit standard samples in lieu of written specifications, and for many diatomaceous products this is recognized as good practice.

It is beyond the scope of this article to give detailed specifications for diatomaceous products. The following may serve as guides in indicating the properties considered most important for different general uses. Insulating compositions: apparent density (brick, block, etc.); bulk density (aggregates and powder); microscopic structure; mesh or screen analysis (aggregates, granules and powders); thermal conductivity; moisture. Filter powders: microscopic structure and particle size; chemical purity and inertness; low density; filter performance. Fillers: chemical purity and inertness; particle size and microscopic structure; fineness and freedom from grit; color; absorptive capacity. Admixtures: reactive silica; chemical composition; fineness; workability.

#### MARKETING, USES AND PRICE

*Marketing.*—The important sources of diatomite are generally distant from the principal markets. Furthermore, the material is bulky, both factors contributing to high freight charges. Packaging costs also are high, for care is required in packing and shipping because of the fragile nature of brick and possible injury to the structure of powders. Diatomite materials are rarely shipped in bulk, although crude may be so handled. The greater tonnage of diatomaceous products is purchased in carload quantities.

Marketing problems and the development of uses in the United States have been largely promoted by the pioneer and largest producer, first the Kieselguhr Company of America, then the Celite Company, and now the Johns-Manville Corporation. The policy of these organizations consistently has been one of progressive research and development (process, product, and uses). The uses of diatomaceous silica, in order of approximate importance, may be classified broadly as follows: filtration, insulations, fillers and miscellaneous, and admixtures. It is not possible here to do more than list some of the very extensive applications and uses.

*Filtration.*—Raw cane-sugar manufacture, cane-sugar refining, beet-sugar manufacture, molasses, corn sugar and glucose, maple sirup, beer, wines, distilled spirits and liquors, fruit juices and beverages (grape, prune, cider, citrus), malt products and extracts, water, dry, cleaning, mineral oils and petroleum products (dehydrating, emulsion breaking, refining, filtration, dewaxing, sludges, tars), vegetable oils (cottonseed, peanut, linseed, corn, castor, coconut), animal oils and fats (fish oil, lard), liquid soaps, gelatin, glue and adhesives (vegetable and animal), chemical solutions, salts and salines, metallurgical slimes and

solutions, sewage, gas purification, pyroxylin and cellulose acetate, varnishes and lacquers, trade wastes and effluents, rigid filters (bacterial etc.), porous diaphragms, separators, etc., flavoring extracts, vinegar, hydrogenated oils, alginates, starch pastes, sizing, etc., dyestuffs, used crankcase oils, analytical and laboratory uses, serums and antitoxins, perfumes, extracts, pharmaceuticals and cosmetics, vitamin extracts, et cetera.

*Insulations.*—Diatomite in its various forms is widely used for industrial heat and cold insulations: power-plant equipment, in steel and nonferrous metallurgy and heat-treating, petroleum refineries, glass manufacture, ceramic kilns and equipment, gas-generating equipment, ovens, furnaces, kilns, pipe-covering, refrigerators, cold-storage buildings, metal-annealing, etc. The common types of products used are block, brick and slabs, aggregates, powders and cements. Brick may be "natural" as cut from the deposit or fabricated with or without inorganic binders and with or without additional porosity as by burning or melting out organic matter. Block and pipe-covering are molded with bonding agents, fiber, magnesia, etc. Cast compositions are made with aggregate or powder and a hydraulic bonding agent such as cement, gypsum magnesia, etc. Mortars, cements, etc., are made up with bonding agents, fiber, etc., and usually mixed with water and applied on the job. For metal-annealing, powder is generally employed. For cold-storage warehouses and low-temperature insulations, block, aggregate or powder is commonly used. In building and home insulation for both heat and sound, powder, granules, blocks, sheets, etc., are standard forms.

*Fillers.*—Absorbent for acetylene, battery-box compositions, molded asphaltic compositions, roofing, flooring, waxes, paints, varnishes, lacquers, pigments, catalyst support (hydrogenation of vegetable oils, coal and oil shale), oxidation and vapor-phase reactions, fuses and explosive caps, rubber, match-head compositions, natural and synthetic plastics, sulphur, dynamite and other explosives (liquid oxygen, etc.), polishes (silver, metal, automobile, cake, liquid, paste), carrier for chemical reactions, fire-resistant compositions, absorbents, insecticide carriers, paper (wallpaper, blotting, filter, glassine, printing, pitch control), abrasives (glass, dental, cleansing compositions), absorbent for radioactive emanations, safe filler, linoleum, artificial leather, sealing wax, glue, artificial stones, pharmaceuticals and cosmetics, putties, packings, etc., to prevent caking and hardening, mold wash, soaps, phonograph records, printing inks, absorbents for disinfectants, drying agent, fertilizers.

*Miscellaneous.*—Manufacture of water glass and soluble silicates, manufacture of ultramarine, manufacture of glazes, enamels, etc., fine reactive silica for chemical processes, manufacture of oil-decolorizing products and synthetic silicates.

*Building Industry.*—Admixture for concrete, mortars and cements; constituent of high-silica cements and puzzolan cements; flooring, pave-

ments, roads, roofing, light-weight building brick, block slabs, etc., wall-board, panels.

*Price.*—Prices for diatomaceous products have remained reasonably constant since 1918, when the increase in production costs due to war conditions was pronounced. The increased costs of fuel, labor and equipment during the period 1920 to 1930 were offset to some extent largely by process improvements and increased operations of the major producers. Cost trends were somewhat lower beginning in 1931. Detailed costs for diatomaceous earth are somewhat difficult to give, since the price range is rather broad, depending on the source of the earth and the processing necessary to prepare it for market. Crude diatomite at the deposits in Denmark, Soviet Russia, Algeria, and in western United States is cheap, \$5 per ton or less, upward. Standard products, admixtures, insulation and filtration grades range in price from \$10 to \$40 per ton at the mines. For highly purified or selected specialty grades, the prices come to \$100 per ton or higher. Insulating brick manufactured in the United States range in price from \$40 to \$80 per thousand at shipping point. Prices for kieselguhr have been listed in *Chemical and Metallurgical Engineering* as follows: 1921–1922, \$61 per ton; 1923–1934, \$50 to \$55 per ton.

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## CHAPTER XIV

### FELDSPAR

By B. C. BURGESS,\* MEMBER A.I.M.E.

**F**ELDSPAR is the commonest mineral in the crystalline rocks. It is usually in small grains intimately associated with other minerals. Commercially, feldspar is obtained only from pegmatites. According to Hess,<sup>21</sup> "The pegmatites are undoubtedly the most bizarre, the most contradictory, the most complex and altogether the most interesting group of rocks known."

#### COMPOSITION AND PROPERTIES

The feldspars form a group of which the principal species are orthoclase, microcline, albite and anorthite. These are aluminum silicates of potassium, sodium and calcium. There are also a barium feldspar, celsian, and barium orthoclase feldspar, hyalophane, rarely found and of no commercial importance. None of the minerals in the feldspar group are found pure or nearly pure. The potash feldspars, orthoclase and microcline, always contain some albite (soda orthoclase and soda microcline, anorthoclase). In turn, the soda feldspars always contain some anorthite (lime feldspar). There is a series of soda-lime feldspars known as plagioclase in which the albite and anorthite molecules replace each other in varying proportions from albite through oligoclase, andesine, labradorite, and bytownite to anorthite. Chemical composition of the principal feldspars is given in Table 1.

Commercial feldspars, in addition to being intergrowths of at least two species of feldspar, always contain one or more of the accessory minerals, such as quartz, muscovite, biotite, garnet and tourmaline, as well as small but varying proportions of the decomposition product, kaolinite. An intergrowth of quartz and feldspar is frequently found containing about 75 per cent feldspar and 25 per cent quartz. The quartz is often distributed through the feldspar uniformly and appears in parallel lines of wedge-shaped grains. This is referred to in the literature as "graphic granite" or *schriftgranit*, and by the miners is called "corduroy" spar.

Perthite is an interlamination of orthoclase and albite. Originally, the name was applied to the mineral from Perth, Ontario, but now it is in

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<sup>21</sup> References are at the end of the chapter.

general use in the adjective form, "perthitic" intergrowth. All commercial potash feldspars are perthitic. Cornwall stone is a feldspar-quartz-kaolinite product obtained from a semikaolinized deposit in England. A similar product from North Carolina is marketed as Carolina stone.

TABLE 1.—*Composition and Properties of Principal Feldspars*

Mineral	Orthoclase	Microcline	Albite	Anorthite
Formula	KAlSi <sub>3</sub> O <sub>8</sub>	KAlSi <sub>3</sub> O <sub>8</sub>	NaAlSi <sub>3</sub> O <sub>8</sub>	CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub>
CHEMICAL COMPOSITION, THEORETICAL PER CENT				
SiO <sub>2</sub> .	64.7	64.7	68.7	43.2
Al <sub>2</sub> O <sub>3</sub> .	18.4	18.4	19.5	36.7
CaO.				20.1
K <sub>2</sub> O...	16.9	16.9		
Na <sub>2</sub> O.			11.8	
PHYSICAL PROPERTIES				
Crystal System	Monoclinic	Trichinic	Trichinic	Triclinic
Refractive index	1.524	1.526	1.529	1.584
Specific gravity	2.56	2.56	2.605	2.765
Melting point, deg. C.	1200	1200	1110	1532

*Properties.*—The crystals of the different species are similar in angle, in general habit (Table 1) and in methods of twinning. The prismatic angles are nearly 60° and 120°. The cleavage is good in two directions at 90°, or nearly 90°. The luster is vitreous to pearly (subvitreous to dull in altered varieties). The streak is white. The fracture is uneven. Colors are generally white, cream and pink, also milky, clear, buff, brown, red, gray, green and bluish. Hardness is 6 to 6.5.

#### ORIGIN, MODE OF OCCURRENCE AND DISTRIBUTION

According to Clarke,<sup>16</sup> the feldspars constitute 59.5 per cent of the igneous rocks, quartz being the next most abundant single mineral at 12 per cent. The hornblende-pyroxene (ferromagnesian) group comprises 16.8 per cent of the average.

Pegmatites, the chief source of feldspar, vary greatly in mineral composition, but as sources of commercial feldspar the "granite" type, consisting essentially of feldspar, quartz and mica, predominates. A second type, according to Bastin,<sup>8</sup> classified as "soda" pegmatite, contains no quartz, no tourmaline, and little or no mica, but consists mainly of albite with small quantities of hornblende. Pegmatites are emanations from granitic magmas that have solidified in dike-like bodies in the enclosed

rocks. Their formation has probably been a long continuing process in which hydrothermal replacement followed the original magmatic injection. They are found in schists, gneisses, granite, diorite and other deep-seated rocks. However, a few pegmatites are found in limestone and other sedimentary rocks. As a natural consequence of such a mode of origin, pegmatites are characterized by extreme irregularity in size and shape of the deposits, size and distribution of the essential minerals, and number and proportion of the accessory minerals. According to

TABLE 2.—*Analyses and Calculated Mineral Content of Commercial Feldspars of the United States and Canada*

PERCENTAGES

No.	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	Loss	Potash Feldspar	Soda Feldspar	Lime Feldspar	Quartz	Other Minerals	Source
1	87.0	18.5	0.07	0.3	T	11.8	2.0	0.3	69.9	17.0	1.5	5.9	5.7	North Carolina
2	87.5	18.3	0.05	0.5	T	10.3	3.0	0.3	61.0	25.4	2.5	8.0	3.1	
3	88.8	17.8	0.1	0.5	T	9.5	3.0	0.3	56.3	25.4	2.5	11.2	4.6	
4	89.5	17.5	0.1	0.8	T	8.1	3.6	0.3	47.9	30.6	4.0	14.1	3.4	
5	73.5	15.3	0.1	0.5	T	7.3	3.0	0.3	43.2	25.4	2.5	25.2	3.7	
6	69.0	18.2	0.1	1.3	T	5.5	5.5	0.3	32.4	46.6	6.5	13.2	1.3	Virginia
7	70.0	18.1	0.1	1.5	T	3.5	6.5	0.3	20.7	55.2	7.5	15.7	0.9	
8	67.3	18.5	0.05	0.5	T	11.0	2.6	0.3	65.1	22.0	2.5	7.1	3.3	
9	69.1	17.5	0.1	1.0	T	9.0	3.2	0.2	53.3	27.2	5.0	12.8	1.7	New York
10	67.6	18.1	0.1	0.3	T	11.3	2.3	0.2	67.0	19.5	1.5	8.4	3.6	
11	74.6	15.3	0.13	1.0	T	2.8	5.8	0.3	16.6	49.3	5.0	26.7	2.4	New England
12	65.4	19.6	0.1	0.2	T	11.3	3.4	0.2	67.0	28.8	1.0	0.4	2.8	
13	67.8	18.4	0.1	0.3	T	10.0	3.0	0.3	59.2	25.4	1.5	8.8	5.1	
14	71.3	16.1	0.1	0.2	T	9.4	2.9	0.2	55.6	24.6	1.0	17.0	1.8	Canada
15	74.0	14.8	0.1	0.3	T	8.1	2.6	0.2	47.9	22.0	1.5	25.8	2.8	
16	65.1	19.5	0.15	0.4	T	13.1	1.7	0.2	77.5	14.4	2.0	1.8	4.3	Maryland
17	65.5	18.7	0.1	0.4	T	12.8	2.3	0.2	75.7	19.5	2.0	1.2	1.6	
18	65.7	19.6	0.1	0.7	T	10.6	2.9	0.3	62.7	24.6	3.5	4.0	5.2	S. Dakota
19	68.2	19.6	0.1	0.7	T	2.1	9.1	0.3	12.4	77.2	3.5	4.3	2.6	
20	64.7	19.7	0.04	0.3	T	12.2	2.8	0.4	72.2	23.8	1.5	0.0	2.5	Colorado
21	66.3	19.4	0.13	0.7	T	9.5	3.6	0.3	56.3	30.6	3.5	5.1	4.5	
22	65.8	18.8	0.04	0.3	T	11.8	3.1	0.3	69.8	26.3	1.5	1.4	1.0	Minnesota
23	65.1	19.7	0.04	0.3	T	12.2	2.4	0.3	72.2	20.4	1.5	1.3	4.6	
24	66.1	19.2	0.1	0.4	0.2	10.7	3.3	0.2	63.4	28.0	2.0	3.3	3.3	California

Schaller,<sup>33</sup> the general sequence of mineral formation in a pegmatite is somewhat as follows: high-temperature potassium feldspar, inversion to microcline, a little perthite, quartz, albite, muscovite, and the general group of such minerals as black tourmaline, garnets and beryl, followed by the lithium minerals, the phosphates, sulphides, carbonates and oxides.

The kaolin content of all commercial feldspars is a result of the natural processes of rock decomposition. Surface weathering undoubtedly is the cause of kaolinization of the feldspars of the Southern Appa-



lachian region of the United States. The kaolin in Cornwall stone may be the result of pneumatolytic action. Carbonic and humus acids are known to be effective agents in the kaolinization of feldspar. Sericitization often accompanies kaolinization. Either or both are often accompanied by solution of iron-bearing minerals, which stain the feldspar.

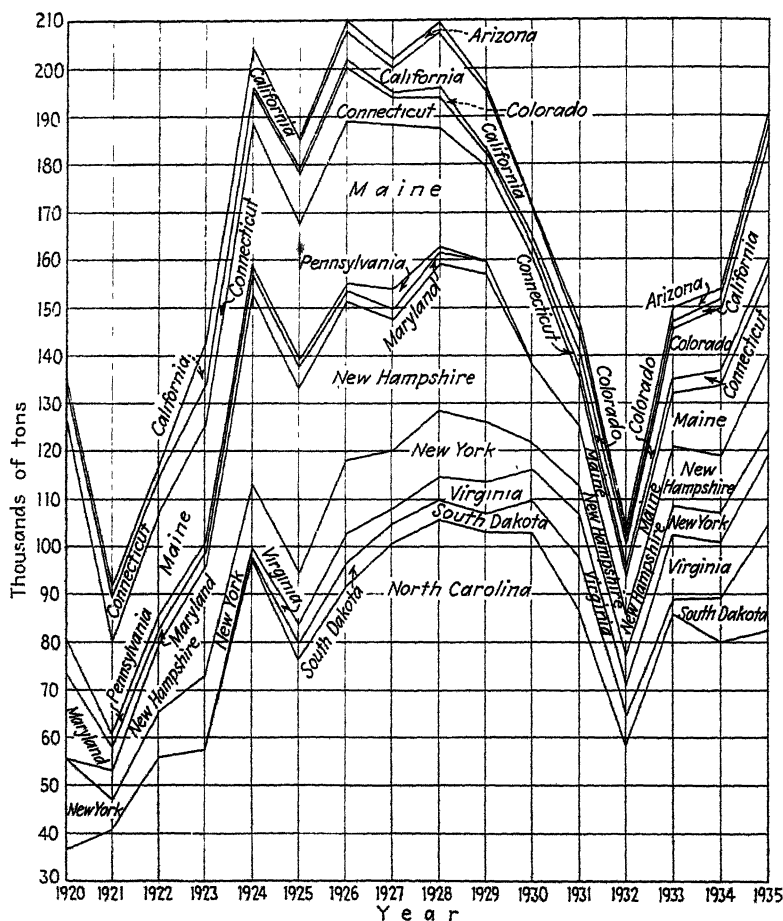


FIG. 1.—CRUDE FELDSPAR SOLD OR USED BY PRODUCERS IN THE UNITED STATES.

Another deleterious impurity is manganese, which frequently is found as dendrite on the cleavage planes of partially kaolinized feldspar.

Analyses of commercial feldspars of the United States and Canada are given in Table 2.

Pegmatites that are present or potential sources of feldspar are found in all the states of the Appalachian Region of the United States from Georgia to New York, and in the New England States. Recently Minnesota, Colorado and South Dakota have become important produc-

ing states. Other western states that are sources of feldspar are California, Nevada, Arizona, New Mexico and Texas. For years Maryland and Pennsylvania were the principal producing states. Maine took the lead in 1913, to lose it to North Carolina in 1916. Connecticut and New York have been consistent producers of 10,000 to 20,000 tons each annually for many years. For the last 10 years North Carolina has averaged about 50 per cent of the total production of the United States. During this same decade Colorado and South Dakota have come up from less than 1000 to 20,000 tons each annually. Production of crude feldspar from 1920 to 1935 is shown in Fig. 1.

Pegmatites containing commercial feldspar are found in the provinces of Canada from Nova Scotia and Labrador to Manitoba, and through the Rocky Mountain area. Most of the Province of Quebec north of the St. Lawrence and the major portion of Ontario consist of crystalline rocks containing many pegmatite dikes. The Scandinavian peninsula has long been the source of most of the feldspar used by European potteries. Andersen<sup>4</sup> reports the examination of specimens of feldspar from upwards of three hundred localities, for the most part situated in southern Norway. Pegmatite dikes are almost equally numerous in Sweden and most productive deposits have been found near Kolsva, Margritelund and Dröm. Pegmatite dikes from which feldspar is or may be obtained are also found in Czechoslovakia, Germany, France, Italy, Rumania, Russia, China, Finland, India, Japan, Australia, Egypt, South Africa and Argentina. No pegmatites that will serve as commercial sources of feldspar are known in the British Isles, but in Cornwall, near St. Austell, large deposits of semikaolinized granite or alaskite are found, from which the product known as Cornwall stone or "china stone" is obtained. A somewhat similar formation is mined at St. Yrieix, France, southwest of Limoges. What would be considered in America a very low-grade pegmatite is mined in Germany for some of the feldspathic material used in that country.

#### POLITICAL AND COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

Owing to the wide distribution of deposits over the world and the comparatively low unit value of the product, feldspar has never entered largely into world commerce. The first porcelain products in which feldspar was responsible for the fluxing action, and for which it is principally used today, were no doubt made from "natural" pottery clays containing the requisite feldspar content. During the Ting dynasty (A.D. 621-945) there is evidence of the first use of feldspar as a separate material for manufacture of vitrified products in China, but it was not until 1709 that porcelain was made in Europe.\* In that year Boettcher, at

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\* C. F. Binns in *The Americana*, The Americana Co., New York, N. Y.

Meissen, Saxony, probably used feldspar in his product. The Ytterby feldspar mine in Sweden is said to have been opened in 1780 and was still reported in operation in 1925.<sup>36</sup>

The most important world movements of feldspar have been as follows: Sweden to European countries, principally Germany, France, Great Britain and Belgium; Norway to European countries, principally Germany, France, Great Britain, and Poland; Czechoslovakia to Germany, Austria, Rumania, and Poland; and Canada to the United States.

*Tariffs.*—The tariff in the United States prior to 1922 on ground feldspar imported was 20 per cent, ad valorem. This was raised to 30 per cent in 1922. No change was made in 1930. Crude feldspar was placed on duty list in 1930 at \$1 per ton of 2240 lb. This was reduced by order of the President in 1931 to \$0.50 per long ton. In Canada crude and ground feldspar entered duty free until May 1930, when a duty of 15 per cent ad valorem was applied to ground spar. This was increased to 30 per cent in 1931.

*Production and Consumption.*—Feldspar production in the United States dates back to the eighteen sixties, following shortly after the establishment of the pottery industry here.\* Between 1880 and 1898 annual production varied between 6000 and 18,000 tons. In 1899 tonnage increased to 27,202. Most of this increase was due to the new use of feldspar in scouring soap. In 1903, production was 41,891 tons; in 1911 it was 92,700 tons; and in 1924 it had increased to 204,772 tons.

Consumption in short tons may be taken very nearly at the same figures as the long tons reported for crude production, the difference of 240 lb. representing approximately the sum of the handling and other losses between crude and ground product. There are also annual differences in accord with the difference in crude inventory at the various mines and plants from year to year. Salient events affecting consumption of feldspar in the United States have been:

1. Beginning of the pottery industry about 1853.
2. First use of feldspar for artificial teeth, in 1883.
3. Discovery in 1896 that alumina is a valuable constituent in glass. (This use developed very slowly, only 1 per cent in 1913.)
4. Discovery of advantage of feldspar as base for scouring soaps, about 1897.
5. Use of considerable quantities of feldspar for poultry grit and roofing granules, which began about 1900. This use reached a peak of

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\* Partly kaolinized feldspar, according to some early records, was mined by the Indians in North Carolina and shipped to England for pottery manufacture as early as 1744. Statistical records are available from the year 1880, but commercial production began at least 15 years earlier, as a mill for grinding flint and feldspar was built at Trenton, N. J., in 1865. In 1883 six states produced a total of 14,000 tons, all of which was used in pottery except a small quantity of high-grade material from Delaware used in the manufacture of artificial teeth.<sup>10</sup>

20 per cent of the total feldspar production in 1920, declining to less than 1 per cent in 1935.

6. Feldspar first used as a binder in abrasive wheels in 1907.

7. During the World War period some high-potash feldspar was used in Portland cement to increase the by-product potash recovery. About the same time a few hundred tons of high-potash spar was shipped from North Carolina to a plant near Atlanta, Ga. for potash extraction by one of the patented processes.<sup>19</sup>

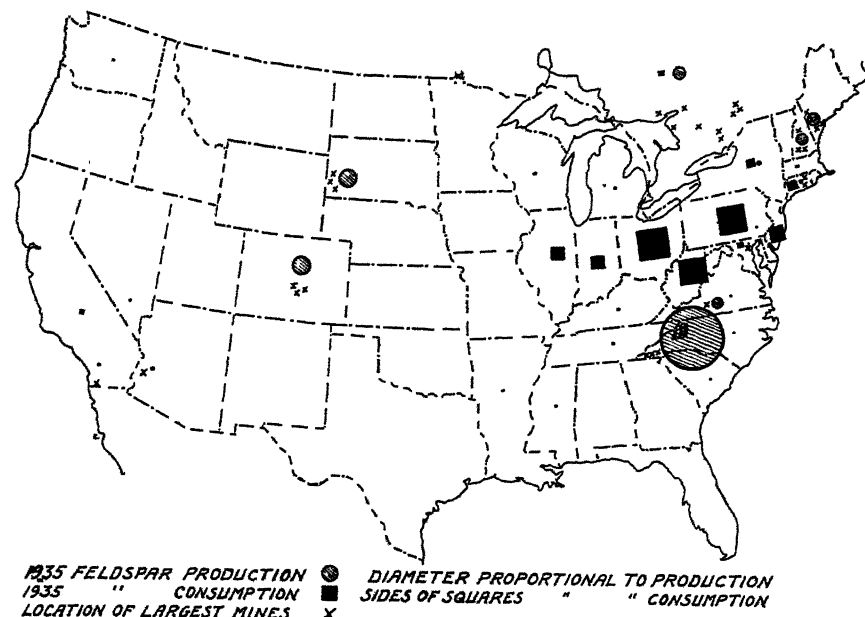


FIG. 2.—FELDSPAR PRODUCTION AND CONSUMPTION AND LOCATION OF LARGEST MINES IN UNITED STATES AND CANADA.

8. Reduction of pottery manufacture as a nonessential industry during the World War.

9. Stimulation of pottery manufacturing, especially floor and wall tile, by the building boom of 1923 to 1926.

10. Use of feldspar in container glass generally adopted by largest manufacturers in 1924.

Production was first recorded in Canada in 1890, when 700 tons of crude feldspar was shipped to the United States. The maximum Canadian production was attained in 1924 with 44,804 tons. Production dropped from 37,527 tons in 1929 to 6921 tons in 1932, and increased to 15,934 tons in 1935. Of this, 85 per cent was exported to the United States in 1924; 80 per cent in 1929; 29 per cent in 1932 and 63 per cent in 1935.

TABLE 3.—*World Production of Feldspar<sup>a</sup>*

Year	Long Tons (2240 Lb.)							Metric Tons (2205 Lb.)			
	1921	1925	1926	1927	1928	1929	1930	1931	1932	1933	1934
United States . . . . .	204,772	185,706	209,989	202,497	210,811	197,699	171,788	149,480	106,396	153,051	156,663
England . . . . .	55,756	57,379	47,769	63,612	61,379	64,558	62,920	60,000	60,000	60,000	60,000
Sweden. . . . .	18,999	26,321	38,434	30,152	39,290	38,475	32,789	33,113	23,693	32,507	34,468
France . . . . .	b	b	18,951	15,000	31,500	b	b	10,700	b	b	b
Czechoslovakia <sup>d</sup> . . . . .	b	b	30,000	30,000	30,000	30,000	30,000	30,000	30,000	30,000	30,000
Canada . . . . .	40,003	25,608	32,099	26,051	23,479	33,506	23,925	16,640	6,393	9,669	16,603
Norway . . . . .	20,530	26,355	20,181	27,213	23,894	26,104	19,608	15,105	13,015	17,986	22,139
Russia . . . . .	b	8,477	20,629	19,019	19,987	b	b	b	b	b	b
Germany . . . . .	32,605	10,093	6,553	7,204	6,132	7,574	5,069	5,000	3,550	4,490	6,808
Italy . . . . .	3,200	2,500	5,785	3,990	4,882	6,700	5,659	4,750	5,217	4,861	7,637
Rumania . . . . .	b	b	1,662	1,912	2,790	2,440	1,932	3,068	681	1,309	1,026
Finland . . . . .	659	788	800	643	700	b	b	67	1,529	2,706	3,329
Argentina . . . . .	b	b	b	183	403	420	193	172	369	376	431
Australia . . . . .	15	32	122	108	161	78	69	209	1,022	2,009	2,948
South America (exclusive of Argentina) <sup>e</sup> . . . . .	b	b	99	b	29	b	b	b	b	b	6,000
Japan . . . . .	23,050	18,743	b	b	b	b	b	b	b	b	b
Egypt . . . . .	b	b	b	b	b	b	b	b	b	b	b
India . . . . .	b	b	b	b	b	b	b	b	b	60	638
China . . . . .	b	b	b	b	b	b	b	b	26,858	27,189	27,780

<sup>a</sup> Production figures for 1924-1930 from Mines Branch *Bull.* 731, Dept. of Mines, Ottawa, Canada, p. 26; for 1931-1934, U. S. Bur. Mines Minerals Yearbooks. Data for China include Manchuria, from Minerals Trade Notes, U. S. Dept. of Interior (1936) 3, No. 1.

<sup>b</sup> Data not available.

<sup>c</sup> Principally Cornwall stone.

<sup>d</sup> Estimated figures for Czechoslovakia and England 1931-1934, inclusive.

<sup>e</sup> Production for 1934 from Chile.

Table 3 shows world production of feldspar for the years 1924 to 1934, inclusive and Fig. 2 shows production and consumption in Canada and the United States, and also the locations of the largest mines.

#### PROSPECTING, EXPLORATION AND MINING

Feldspar has been found outcropping plainly at the surface in so many parts of the world in reasonable proximity to consuming areas that no systematic methods have been required for prospecting and exploration. In pegmatite-bearing areas new deposits have repeatedly been found by crude methods involving for the most part only hand labor. A great deal of labor, however, if not so much money, has been wasted by the failure of prospectors and miners to use the most elementary principles of mining in opening up deposits. Watts called attention to this in 1916.<sup>40</sup>

Two or three companies have tried core drilling as a means of testing the extension of deposits. The results have not entirely justified the expenditures, because the average deposit is small and the distribution of its commercial feldspar content is extremely irregular. Exceptions are the large and comparatively uniform deposits in the western part of the United States. In most of these deposits surface indications furnish sufficient information as to size, extent and quality, so drilling is unnecessary.

There is a theory supported by a number of writers on the subject that good feldspar deposits are generally hidden by a comparatively shallow capping of mixed pegmatite material. As a result thousands of dollars have been wasted on pegmatites that a mining engineer experienced in development of feldspar would never have considered working. No better advice can be given than to work only where one finds plenty of float material or a good outcrop and to continue to work only as far as the feldspar continues. "Sticking to the ore" in the development of a mine is a maxim as old as mining itself, and it pertains to feldspar as well as to the more valuable minerals to which it is generally applied. Following this procedure, what will be the source of feldspar when all surface outcrops have been exhausted? The probability is that lower grade pegmatites still outcropping will be utilized by beneficiation methods before risks are taken to find buried deposits of the present quality.

The great majority of feldspar mines are open-cut or quarry operations and the methods used are so common to that type of operation that no description is necessary. In the United States and Canada the topography of all the mining districts is hilly or mountainous. A cut is run in or a pit sunk in good float material or mining is started directly on an outcrop. Hand drilling is done until the openings, together with surface indications, show sufficient feldspar to justify the expense of moving in a portable compressor and air drills or running a pipe line from the nearest mine.

Material is moved out by wheelbarrow, car line, sled or motor truck. It is hoisted by windlass, whim or derrick or by hoist and derrick. Discarded automobiles have been the boon of the feldspar miner. An old car obtainable for between \$10 and \$50 provides an engine suitable for hoisting, pumping or air-compressor drive. Some of these cars are suitable for haulage purposes, usually for waste disposal, and often are operated at a cost saving, even in the South where labor rates are low. At the comparatively few large feldspar-mining operations more expensive equipment is installed. Stationary power plants and railroads and aerial tramways have been built at some mines. With the advance in efficiency of the portable compressors and economy of operation of motor trucks on grades up to 20 per cent or more, it is probable no other equipment will be justified in the future even at the largest mines.

The mine of the Golding-Keene Co. at Gilsom, N. H., illustrates work on the larger type of deposits. A mica mine had been worked for many years along what was known to be the hanging wall of a large pegmatite. As exhaustion of the mica on that wall approached, it was decided to crosscut to the footwall. Almost pure feldspar was encountered for a width of about 200 ft. Ladoo<sup>26</sup> describes the development. It is interesting to record that even with the extensive exploratory workings at this deposit the effort made to project the extension of the feldspar a comparatively short distance beyond its developed limits proved in error and an inclined shaft sunk at considerable expense was abandoned after encountering only fine-grained pegmatite, from which practically no feldspar could be sorted. The operators then went back to the feldspar in the crosscuts, raised to the surface and opened out a bottle-shaped pit. This has now attained a depth of over 200 ft., is about 150 ft. wide and 300 ft. long.

The Deer Park mines comprise a group of five deposits in a horseshoe bend of the South Toe River, about three miles below Spruce Pine, N. C. No. 5, the largest, in the center of the group, is about 100 ft. wide and has been worked along the strike for a distance of 300 ft. at depths principally from 100 to 250 ft. The greater portion of the mining at Deer Park No. 5 has been done below the zone of weathering and the rock is all hard and tight. Although the workings extend almost 200 ft. below the bed of the river, there is no seepage. The main portion of the ore has been removed by room-and-pillar methods and sorted on the surface or on picking belts at the plant, one mile up the river. Where the dike forked out into the country rock, a cut and fill and a shrinkage stope were run. Hoisting is done from two different levels in Joplin-type "cans" or buckets trammed on four-wheeled trucks. Production has averaged about 1 ton of spar and 32 lb. of mica to 3 tons of waste.

Mica mentioned in the description of both of the mines is an important by-product of some feldspar operations. It is found in "wedge"

or "flat" blocks, the latter being much the more valuable of the two. Quartz is also occasionally a salable by-product.

After the deposit has been prospected, opened up and equipped for mining, the most important part of the operation is cobbing.\* A few deposits have been found containing feldspar clean enough so it could be shipped just as it is blasted from the mine, but these are exceptions. In the great majority of cases, after blasting the lumps must be broken by hand with hammers to remove quartz, mica, garnet or other minerals.

Where it is not practical to remove the quartz, grades are made containing different proportions of this mineral, but practically all mica, garnet and other minerals must be removed from all grades of crude feldspar. In mines containing both soda and potash spar, these are generally separated. A separation is also made as to soft (semikao-linized) and hard spar.

Interesting old mines still in operation in the Stockholm district in Sweden are described by Spence.<sup>36</sup>

#### PREPARATION FOR MARKET

The practice in Europe is to ship the crude spar to mills in the consuming centers, where it is ground, usually by wet methods, and used at adjacent manufacturing plants or delivered by truck to the neighboring consumers. In the United States and Canada all feldspar is ground dry. Of the 30 plants now operating in the United States, 4 are in consuming centers; 17 are at the mines and 9 are at intermediate points where milling-in-transit rates or their equivalent are enjoyed. Prior to 1916 there were a few jaw crushers in use but most of the feldspar was sledged by hand small enough to feed to chaser stones. This product was loaded in batch pebble mills and ground for a period of time that had been found necessary to reduce it to the required fineness.<sup>40</sup> Air separation, introduced in 1916, revolutionized milling methods, making possible continuous grinding.<sup>13</sup> The modernization that resulted quadrupled the grinding capacity of the industry. "Chemical control," for which a plant<sup>14</sup> was first constructed in 1926, made it possible to produce uniform grades of feldspar and relieved consumers of their most serious problem concerning raw materials. The next advance in milling came in 1930 with the production of a special granular product for the glass industry, made possible by development of high-intensity magnetic separation.<sup>15</sup>

Fig. 3 shows a detailed flowsheet of one of the two largest plants in the industry. This plant of the Tennessee Mineral Products Corporation, at Minpro, N. C., and the plant of the Consolidated Feldspar

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\* "Upon the care exercised in cobbing, sorting and grading depends the entire success of a feldspar-mining undertaking, and lack of enough knowledge or skill in these operations, or neglect to conduct them properly, must always cause trouble and invite failure." [J. L. Stuckey, former State Geologist of North Carolina in *Manufacturer's Record* (June 1927).]





Corporation at Erwin, Tenn., are the only two equipped for production of granular spar and magnetic separation. The flowsheets of the two are similar, except that the Erwin plant uses two Symons cone crushers for pulverizing in place of four sets of rolls at the Minpro plant. Some of the smaller plants produce a semigranular product by removal in part of the fines by air separation from the regular 20-mesh pebble-mill product.

Table 4 summarizes the fine-grinding equipment in the industry, July 1, 1936. It also gives the average production rates in grinding the three principal sizes, 20 mesh, 100 mesh and 200 mesh. From these and the relative consumption of these sizes, capacity of the industry has been calculated and shown in the lower portion of the table. These capacity figures may be considered excessive because they do not provide for peaks of consumption above the average that all industries must absorb. However, making 20 per cent reduction for such peaks, the total still represents more than three times the maximum consumption of ground feldspar in any year to date. Table 5 gives other production figures.

TABLE 4.—*Summary of Grinding Equipment and Capacity Based on Proportion of Different Sizes, 1931-1935*

Number of Mills	Type	Continuous or Batch	Average Size	Average Grinding Rate		
				20 Mesh	100 Mesh	200 Mesh
43	Conical.....	Continuous	8' X 48"	4.00	3.00	1.50
5	Cylindrical.....	Continuous	8' X 10'	3.50	2.50	1.30
19	Tube <sup>a</sup> .....	Continuous	5' X 22'	b	2.00	1.25
38	Cylindrical.....	Batch	7' X 6'	b	0.82	0.42

CALCULATED CAPACITY

Mesh	Per cent Consumed	Total Capacity, Tons	Time Ratio	Time Per cent	Average Capacity, Tons
20	47.17 <sup>c</sup>	1,234,800	29.19	37.88	467,742
100	10.81 <sup>d</sup>	1,409,760	5.85	7.59	107,001
200	42.02 <sup>e</sup>	763,992	42.02	54.53	416,605
100.00		100.00			991,348
Capacity/ two plants equipped for granular and semigranular production					86,400
Total grinding capacity/ of the industry.....					1,077,748

<sup>a</sup> A cylindrical mill with length two or more times diameter.

<sup>b</sup> Not practical for 20-mesh (coarse) grinding.

<sup>c,d,e</sup> Proportions respectively of glass, enamel and pottery feldspar.

<sup>f</sup> Capacities based on 24-hr. operation for 300 days.

The wet grinding practice in Europe until about 1920 was practically the same as has been used since the beginning of the industry. It consisted in feeding 1 to 1½-in. crushed feldspar into grinding pans or

pan mills 12 to 14 ft. in diameter, having floors of hard china stone or granite blocks. Grinding is effected by forcing through the charge of material large stones attached to radial arms. Enough water is added with the charge to make a thin sludge. After this batch is ground the required period, it is washed out through a launder to micas (a series of troughs) and thence to settling pits and storage.

Since the war, pebble mills have been introduced in a few plants. All are run wet and most as batch mills. A few conical mills are in use in closed circuit with rake or bowl classifiers. During this period a new type of classifier has been successfully used with batch mills of both the pan and cylinder type, which may be used for continuous grinding also. It is known as a "Sy-Vor" classifier and operates on the siphon-vortex principle.<sup>5</sup> These are said to be producing to specifications such as all minus 180-mesh, 16 to 17 per cent plus 600-mesh and 50 per cent minus 1000-mesh.

Magnetic separation was first used in 1930. Primarily it was introduced to remove the metallic iron resulting from the special equipment necessary to make a granular product. It was found, however, that iron-bearing minerals such as biotite, garnet, tourmaline and some muscovite were also removed. One plant in the United States is now commercially separating these minerals from run-of-mine pegmatite. A small plant in Canada is making a similar separation of nepheline syenite, producing a nepheline-feldspar product for the glass industry.

Experimental work is being actively carried on with a view to separating quartz and feldspar by froth flotation. Electrostatic separation was applied to feldspar for removal of mica in Bristol, Tenn., in 1922. Although successful, it did not appear warranted at the time. Since then it has been successfully applied to the separation of quartz and kyanite. Now, no doubt, electrostatic separation will receive attention as a method of making a quartz-feldspar separation.

In Czechoslovakia a run-of-mine pegmatite is separated by air selection and screening methods to provide a feldspar product for ceramic use. The separation of mica is effected by passing the oversize product from pebble-mill air separator through a series of burlap screens, which catch the mica and permit the feldspar to pass through. Quartz is removed by taking out of the air-separator circuit certain sizes that have been found to contain quartz segregations. A reduction of silica from 78 to 73 per cent is made.

#### TESTS AND SPECIFICATIONS

Bowles and Justice<sup>10</sup> record the first movement toward standard specifications for feldspar with the classification of grades at No. 1, No. 2 and No. 3 in 1906. However, as they say, "little progress in grading was made for many years. Most consumers designated the kind of spar they

desired simply by its geographic source." The first chemical laboratory in the industry was installed at the plant of the Tennessee Mineral Products Co. at Bristol, Tenn., in 1923. Beginning with this, serious efforts were made toward production of spar of uniform chemical composition and definitely limiting the content of impurities. This was the beginning of chemical control in the industry.<sup>23</sup>

Early in the discussion at the first meeting of the Feldspar Grinder's Institute, July 15, 1929, it was agreed that standardization of the product should be the first objective. This was accomplished through cooperation of the Bureau of Standards Division of Trade Standards and became effective Sept. 1, 1930, as Commercial Standard CS 23-30. It has been so frequently reprinted, and is so readily obtainable\* that it will not be given here. Its essential features are: (1) Physical classification based on fineness of grinding; (2) chemical classification based on composition as it influences use; (3) standard methods for making screen tests; (4) standard methods for making complete chemical analysis.

The Feldspar Code of the N.R.A. provided for standardization without reference to CS 23-30. The miners and some of the small grinders brought together under the N.R.A. refused to agree to this standardization, which to them appeared very complicated. Several attempts were made by the Code Authority to formulate a new standard. The N.R.A. went out of existence before anything was accomplished, but the Feldspar Association has continued these efforts. It has published, under date of Dec. 4, 1935, a modification of CS 23-30, which may be used by producers unable or unwilling to comply with the Commercial Standard. Principal differences are a classification based on use, rather than analysis, elimination of alkali ratio, and 3 per cent difference in grades as to silica content, instead of 2 per cent.

Disregarding the greatly improved feldspar products on the market since chemically controlled blending has become standard practice, a number of consumers have still maintained a prejudice against blending. This attitude should disappear as a result of the recent publication of the paper by Schramm and Hall.†

CS 23-30 does not give a method of fusion test, although these tests are usually made by producers and consumers. The omission was made because no method of making such tests has been found generally acceptable. Some consumers use the block test,‡ and others use the Du Bois test.<sup>20</sup> But the majority of the producers and consumers of feldspar fill

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Superintendent of Documents, Washington, D. C. 5¢ per copy.

† Quoting from conclusion: "The results obtained lend full support to chemical control . . . In the natural intermediate feldspars we have solid solutions of one feldspar in the other and perthitic intergrowth of these solutions. Such structures are not resolved by the grinding process. It is, therefore, apparent that these mineralogical differences are without significance in the use of feldspar as a ceramic flux."<sup>24</sup>

‡ E. P. & Mfg. Co., Trenton, N. J. Pat. 1926.

a small cup or mold a cone of feldspar powder, generally using gum tragacanth or some similar organic binder, and subject this sample to the desired heat in a test or commercial kiln. The color and fusibility of such tests are only of value for comparison with other samples tested under

TABLE 5.—*Sales of Ground Feldspar*

Use	Sales, Per Cent		
	1913	1930	1935
Glass ..	1	30	49
Enamel . .	9	9	9
Pottery..	76	50	37
All other <sup>a</sup>	14	11	5

<sup>a</sup> Includes scouring compounds, roofing and cement-facing granules, poultry grit, cement manufacture (for by-product potash recovery) and abrasives. Dental porcelain (false teeth) and the use as a ceramic binder for abrasive products is included under "pottery."

TABLE 6.—*Formulas of Feldspar Products*

## PERCENTAGES

	Hotel China	Floor Tile	Wall Tile	Sheet- iron Enamel	Elec- trical Porcel- lain	White- ware Glaze	Con- tainer Glaze	Flat Glass
Feldspar ..	17 0	55.0	11.0	30.0	35.0	44.4	15 2	10.34
Flint	37 0	10.0	33.0	20 0	25.0			
Ball clay .	20 0	15.0	27.0		20.0			
China clay	25 0	20.0	29 0		20.0	4 3		
Magnesium carbonate	0 5							
Whiting	0 5					20 4		
Borax..				26.0				
Soda ash..				4.0			16 2	20 74
Soda niter				3.0				
Fluorspar .				3.0				
Cryolite				11.0				
Antimony oxide				3.0				
White lead. . .						24 8		
Zinc oxide . .						6 1		
Sand... .							47.6	51.80
Limestone . . .							18.1	15.55
Salt cake . . .							0 47	1.57
Copper sulphate							2.43	

identical conditions. A complete chemical analysis furnishes a better guide as to the fluxing action of a feldspar than any fusion test so far devised.

Considerable microscopic work<sup>8,22,38</sup> has been done on feldspar with a view to determining more rapidly the constituents than can be done

chemically, but none are in general use. Microscopic work has also been done to determine fine particle sizes,<sup>2,30</sup> below the sizes readily obtained with the finest commercial testing sieve (325 mesh). Other methods of determining fine particle sizes are by means of the Andrews elutriator,<sup>17</sup> the sedimentation method,<sup>9</sup> and an air-analyzer method devised by Roller,<sup>32</sup> soon to be published by the American Ceramic Society.

### MARKETING, USES AND PRICE

*Marketing and Uses.*—The factors involved in the marketing of crude spar are readily determinable only in the districts where there is a competitive market for the products, as in North Carolina. However, there is a definite relation between miners wage rates and the cost of crude spar, wherever it is found. The other principal factor in the cost and marketing of crude spar is the percentage of recovery from the rock broken.

Generally speaking, it takes two man-hours to move a ton of rock in feldspar mining. Labor averages about 50 per cent of the cost. Therefore, in a district where the recovery is only 20 per cent and the wage rate 30¢ per hour, average cost of crude should be \$6 ( $5 \times 2 \times \$0.30 \times 2$ ). In another district where the recovery is 80 per cent and wage rate 50¢ per hour, crude feldspar will cost only \$2.50 per ton. Illustrations of such differences in cost and resulting marketing problems are found in the literature.<sup>43,46</sup> It is evident that if such conditions continued indefinitely the business would soon all go to the low-cost areas. That this has not happened is accounted for by other conditions such as higher transportation costs, quality differences, or rapid reduction in the percentage of recovery as the best segregated pegmatites become depleted in a new district.

With the tremendous capacity for production of ground feldspar in excess of consumption, marketing has become more a problem of selling service than of material available. As a result, the personal relations between buyer and seller are more important than large plant investment or extensive reserves of crude feldspar. The relative importance of the various markets through the last 22 years is well illustrated in Table 6.

Formulas of products in which feldspar is used vary greatly.<sup>42</sup> Those given in Table 6 are selected to show the proportion of feldspar used.

Railroad freight averages 20 to 30 per cent of the delivered cost of ground feldspar in the United States, so freight rates are an important marketing factor here. Mr. Charles Donley, Traffic Representative of the Feldspar Association, has furnished the following memorandum of the present (July 1936) basis of freight rates on feldspar in the United States:

The rates are based on a percentage of the key or base rate of first class. This so-called key or first-class rate is based on mileage and is different in the various sections of the country.

In the territory of high-density traffic, the first-class rate is lower per mile of railroad than in low-traffic density territory. The feldspar rail freight key rate is higher per mile of railroad from North Carolina to New Jersey than it is from Maine to New Jersey. The present rail freight rate from North Carolina to New Jersey or Ohio, as illustrative, is based upon 16 per cent of the key (first-class) rate while from Maine to New Jersey or Ohio the rates are based upon 21 per cent of first class. First class is a base of 100 per cent and feldspar, as the territory runs, is assessed 16 or 21 per cent of the 100 per cent rate.

West of the Mississippi, the present rail freight rates are much lower per mile of railroad than in the East. Some rates in the West, when reduced to the mileage basis are as low as 11.5 per cent of first class.

In the past, Cornwall stone and similar semikaolinized feldspathic materials have been the only products that have seriously replaced feldspar. More recently talc has been substituted for some of the feldspar in semivitreous pottery and tile. Now nepheline syenite is being introduced as a source of alumina in glass, replacing feldspar. Clay, kyanite and pyrophyllite have been tested with a view to similar use.

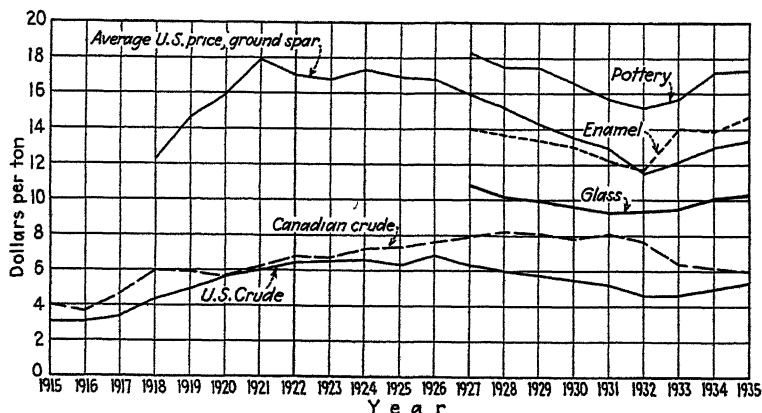


FIG. 4.—AVERAGE VALUE OF CRUDE SPAR SOLD TO GRINDERS, AND SELLING PRICE OF GROUND SPAR.

It appears improbable that any of the substitutes for feldspar so far used or considered would make serious inroads into its established markets as the principal ceramic flux and source of alumina in glass. Expansion of the feldspar market is dependent upon increased manufacture of the principal products in which it is used, pottery, enamel and glass, increased use of these products, and new uses. Pottery and enamel manufacture follow closely the trend of building construction. Flat glass follows the same trend. Container glass climbed to a new peak following "repeal." There is a suggestion of possible increased use of feldspar in pottery through colloidal preparation of slip. Educational work in the glass industry is tending toward appreciation of value of alumina content in flat as well as container glass and increase of alumina in both. If the maximum alumina now thought desirable is attained, it will double the

present use of feldspar in this industry. There are no prospective new uses of feldspar comparable to the markets now supplied.

*Prices.*—The average prices of both crude and ground feldspar have followed the general trend of commodity prices, about doubling in the last 20 years with low points at the beginning of the World War

TABLE 7.—*One Company's Price List<sup>a</sup>*

GLASS FELDSPAR

Grade	20-mesh	Semigranular	Granular	40-mesh
16	\$11 00	\$11 00	\$11 75	\$11 50
17	11 75	11.75	12 50	12.25
18	12 50	12 50	13.25	13 00
19	13 25	13 25	14 00	13.75

ENAMEL FELDSPAR

Grade	100-mesh and Coarser	Finer than 100-mesh
70 per cent and higher silica	\$14.00	\$15.00
Less than 70 per cent silica	15.00	16 00

POTTERY FELDSPAR

	200-mesh and Coarser	230-mesh	325-mesh
All grades	\$17.00	\$18.00	\$19 00

GLAZE FELDSPAR

	\$19.00	\$20 00	\$21 00
All grades			

Above prices per ton of 2000 lb. in bulk carloads, f.o.b. Spruce Pine, N. C. In paper bags, \$1.00 per ton extra. Less carload, minimum 2 tons, \$3.00 per ton advance; less than 2 tons, \$30.00 per ton plus paper-burlap bags at 12¢ each. All above grades and grindings (meshes) according to CS 23-30 except semigranular and granular, which have been added as follows: Semigranular, less than 1 per cent residue on 20-mesh and minimum 62 per cent and a maximum of 85 per cent residue on 200-mesh; granular, less than 1 per cent residue on 20-mesh and a minimum of 85 per cent residue on 200-mesh standard testing sieve.

<sup>a</sup> Tennessee Mineral Products Corporation (subsidiary of United Feldspar Corporation), New York, N. Y.

and in 1932 and a peak at the top of the building boom in 1926. Fig. 4 shows the price history through the last 20 years. The average prices of glass, enamel, and pottery grades are shown only for the period during which the Feldspar Association has been in existence, and such statistics are available. The present complete price list of one company is given in Table 7.



The outlook for the future is that prices will gradually work lower with increasing efficiency in production, probably through beneficiation.

Prices of crude and ground spar in the various countries have not resulted in any considerable foreign commerce except between the European countries, where the situation corresponds more nearly to the movement between states in this country and the importation of Canadian spar to the United States. The latter at its height was caused by the lack of comparable quality in the United States and reluctance of potters to change their formulas.

#### PATENTS

The following is a list of the principal United States patents issued in the last 20 years relative to feldspar:

No. 1310939. Patented July 22, 1919. George J. Bancroft: Process of Separating Mica from Feldspar.

No. 1404974. Patented Jan. 31, 1922. Frank P. Knight and John T. Shimmin: Method of Separating Feldspar and Quartz.

No. 1744967. Patented Jan. 28, 1930. F. R. Johnson: Art of Separating and Apparatus Therefor.

No. 1855115. Patented April 19, 1932. Charles H. Peddrick, Jr. and Philip Wager Lewis, Assignors to United Feldspar Corporation: Process for the Preparation of Feldspathic Flux for Porcelain (The "Chemical Control" Patent).

No. 2007742. Patented July 9, 1935. Leslie Brown: Process of Making Ceramic Roofing Granules.

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## CHAPTER XV

### FLUORSPAR AND CRYOLITE\*

By ERNEST F. BURCHARD,† MEMBER A.I.M.E.

**F**LUORSPAR is one of the nonmetallic minerals of moderate intrinsic value but of great commercial, if not even strategic, importance, because of its usefulness in the metallurgic, ceramic and chemical industries, especially in the manufacture of open-hearth steel, enamel, glass, hydrofluoric acid and refrigerants.

The mineral fluorspar or fluorite, calcium fluoride ( $\text{CaF}_2$ ), contains 51.1 per cent calcium and 48.9 per cent fluorine. The mineral itself is rarely contaminated with impurities but generally is associated with other minerals from which it has to be separated for commercial use. It is a lustrous, glasslike mineral, generally translucent to transparent; it may be clear and colorless, or range in color from slightly bluish through varying shades of violet, amethyst, purple, green, and yellow. It crystallizes in the isometric system and possesses perfect octohedral cleavage. Commonly it occurs in masses of very pure crystalline material with aggregates of cubical crystals in the open spaces, but also is found in fine to coarse granular form. Banded veins and masses with fibrous, radiating structure occur locally.

The specific gravity of fluorspar is 3.18, the hardness is 4 in the Mohs, scale, compared with 7 for quartz and 3 for calcite. It is distinguished from calcite by its failure to effervesce with dilute hydrochloric acid. Heated with sulphuric acid, it gives off fumes of hydrofluoric acid, which etch glass.

Fluorspar is a rock-forming mineral that occurs in various associations. It is most abundant in veins and in sedimentary formations, but also is found as a minor accessory in granite, syenite, pegmatite, gneiss and schist, and more rarely in volcanic rocks. Lindgren<sup>25</sup> regards it as a "persistent mineral" or one of a group of minerals, few in number, that are less sensitive than others to physical conditions and recur under very different conditions. Such minerals are generally of simple composition,

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do not contain the hydroxyl molecule, and include quartz, hematite, pyrite, chalcopyrite, fluorite, calcite and native gold. Fluorite is a common primary gangue mineral of ore deposits and their wall rocks, including those formed by magmatic segregation, contact metamorphism, and deposition in veins from circulating hydrothermal solution at various depths and temperatures. It is also found in sedimentary rocks, and, with galena and sphalerite, in deposits formed at moderate or shallow depths, either by solutions from a deep-seated source, as believed by some, or by descending meteoric water, as contended by others. Deposits in sedimentary rocks include the large deposits of the Illinois-Kentucky district in the United States, those of Durham and Derbyshire in England, and in the Madoc district, Ontario, Canada.

Minerals commonly associated with fluorspar are calcite, barite, quartz, galena and sphalerite. The most important geological features to be considered in connection with fluorspar deposits are structures and source rocks. Most vein deposits occupy fault fissures; deposits in sedimentary beds are generally due to replacement influenced by local structure and favorable composition and texture of the enclosing rocks. Deposits in veins in igneous rocks are readily explained but for those in sedimentary rocks a magmatic source is often sought or inferred.

#### DISTRIBUTION OF DEPOSITS

Fluorspar occurs on every continent and in most of the following countries are deposits that have yielded commercial production: United States, Canada, Newfoundland, Mexico, Guatemala, Brazil, Bolivia, Argentina, England, Scotland, France, Germany, Spain, Italy, Switzerland, Norway, Russia, China, Chosen, Southern Rhodesia, Union of South Africa, Southwest Africa, and New South Wales, Queensland, South Australia and Victoria in Australia.

#### UNITED STATES

Fluorspar is found in several of the Eastern States, but in comparatively few of them are the deposits of commercial value. The largest bodies thus far discovered in the country are those of Pope and Hardin Counties, Illinois, and Crittenden, Livingston and Caldwell Counties, Kentucky, comprising the well-known Illinois-Kentucky fluorspar field. Deposits of minor importance have been worked in central Kentucky, central and eastern Tennessee, and New Hampshire. A number of other localities are known in the Eastern States but from none of them has there been a sustained production.

*Illinois-Kentucky District.*—The Illinois-Kentucky district is probably the greatest fluorspar-producing area in the world. The district, about 40 miles square, comprises the greater parts of Hardin and Pope Counties

in Illinois and of Crittenden, Livingston and Caldwell Counties across Ohio River in Kentucky.<sup>3,4,13-16,20,21,23,35</sup> The area is underlain by nearly horizontal Mississippian limestones, sandstones, and shales, and is characterized by a large number of steeply dipping normal faults, and by the presence of dikes of much altered mica-peridotite and lamprophyre. The fluorspar deposits are of two types: (1) nearly vertical veins occupying fault fissures, and (2) horizontal tabular, or lenticular, deposits.

The vein deposits, typically developed near Rosiclare, Ill., and Marion, Ky., cut sharply through the local formations and in general exhibit a cleaner and firmer footwall than hanging wall. The amount of displacement along the faults varies from a few feet to as much as 1300 ft., but the largest deposits do not occur in faults of greatest displacements. The Rosiclare fault has been traced for about  $4\frac{1}{2}$  miles along the strike and the vein formed in it has been mined underground for a distance along the strike of about 9000 ft. The veins vary in width from a fraction of an inch to 30 ft., and fluorspar has been found to depths of more than 720 ft. in Illinois and more than 400 ft. in Kentucky. The dominant minerals in one of the typical Illinois veins are calcite and fluorite, with quartz, galena, sphalerite, pyrite and chalcopyrite also as primary minerals, and barite, gypsum, malachite, cuprite and others as secondary minerals. In places the fluorspar diminishes and is succeeded by calcite at depth. The vein material was probably formed through two processes of deposition; namely, vein filling and the replacement of brecciated calcareous material and walls, particularly the hanging wall of the fissure.

The tabular, horizontal deposits, variously termed "blanket" deposits, "bedded" deposits, and "bedding" deposits, near Cave In Rock, Ill.,<sup>14</sup> have attracted attention in recent years because of the high quality of the product, the relatively low costs of milling, and the potential reserves. The ore crops out just below the basal shale of the Rosiclare sandstone and dips gently to a probable depth of 200 ft. or more. It consists of banded light and dark fluorspar that has replaced the upper beds of the Fredonia limestone. In places at the top there are a few inches to 2 ft. of coarse-grained, massive, rather pure fluorite. The thickness of the ore ranges from a few inches to more than 12 ft., and the average mined thickness is about 4 ft. Vugs that are found in this zone have yielded many handsome aggregates of well crystallized fluorspar. Recent studies<sup>16</sup> show that the mineralized beds are replacements of certain beds of limestone and are genetically connected with minor fissures, of little or no displacement, that are greatly constricted, or die out entirely at the ore horizon. The shale roof evidently acted as a barrier to the mineralizing solutions that arose along these fractures and spread out beneath the impervious stratum. The Cave In Rock locality lies just

east of the area of pronounced faulting wherein the vein deposits of fluorspar have been formed. The mineralizing solutions may have migrated laterally from major faults through minor fissures. Similar deposits are reported near Carrsville, Livingston County, Ky. The bulk of the fluorspar production has come from the vein deposits, which have produced more than 2,500,000 tons, of which more than 1,000,000 tons has come from the Rosiclare vein to date. More than 100,000 tons has been produced from the bedding deposits, and geological conditions seem favorable for a reserve of many times this output.

*Western States.*—Fluorspar occurs in most of the states from the Rocky Mountains westward. Many of these occurrences are in association with other mineral deposits and are not in themselves of commercial value, but commercial production of fluorspar has been reported from Arizona, California, Colorado, Nevada, New Mexico, Utah and Washington.<sup>7,26</sup> In Arizona<sup>1</sup> occurrences are reported in seven counties, but production has come principally from the Castle Dome district, Yuma County, where, accompanying galena, it occurs as a vein material in volcanic rocks. In California fluorspar occurs, notably in San Bernardino County. Production is reported from near Afton, where the spar occurs in brecciated andesitic rocks. Fluorspar occurs in many places in Colorado<sup>2</sup> as a gangue with metalliferous ores and has been mined on its own account at several places, including Wagon Wheel Gap, Jamestown, North Gate, and Brown Canyon. The spar was used in a small way in refining gold and silver but the basic open-hearth steel plant at Pueblo was the first large market for Colorado fluorspar, and recently some has gone as far east as Pennsylvania. In Nevada the production has come from near Broken Hills, Mineral County, and Beatty, in Nye County.<sup>23</sup> In southwestern New Mexico numerous small deposits have produced fluorspar. Two types of deposits have been recognized, vein fillings in igneous and sedimentary rocks, and blanket replacements in limestone. The veins in Sierra and Dona Ana Counties indicate mineralization to depths of several hundred to possibly 1000 ft.<sup>22</sup> and indicate large reserves of spar, although some of it is siliceous and requires thorough concentration to yield a marketable product. In Utah some fluorspar has been shipped from Beaver and Tooele Counties. In Washington fluorspar has been mined from a vein cutting pink granite near Keller.

#### FOREIGN COUNTRIES

*Canada.*—Fluorspar occurs in British Columbia, Ontario, the Northwest Territories, Quebec, New Brunswick and Nova Scotia.<sup>21,26,36</sup> In British Columbia the "Rock Candy" deposit in the Grand Forks mining division forms a large ore shoot containing a network of replacement veins varying from a few inches to 30 ft. in width, lying within a body of syenite

of Tertiary age. The veins contain fluorspar, barite, chert, crystalline quartz, calcite, pyrite and kaolin, but galena and sphalerite are absent. The product has been used principally at the Trail, B. C., smelter, which consumed also fluorspar from near Keller, Wash., in the early 1920's. Fluorspar occurs in more than 100 localities north of the Great Lakes from Fort William eastward to Ottawa.<sup>36</sup> The "spar" is associated with a large variety of vein minerals and the wall rocks range in age from pre-Cambrian crystallines to Paleozoic limestone and dolomite. Few of the deposits have been of commercial value. Between 1916 and 1920, under the stimulus of war prices, some deposits were opened near Madoc and considerable "spar" was produced.

*Newfoundland.*—High grades of fluxing and acid fluorspar are being shipped from a deposit virtually on tidewater on Little St. Lawrence Bay.<sup>21</sup>

*Mexico.*—Fluorspar occurs in numerous localities in Mexico, associated with metalliferous minerals. It is mined at Monte Realejo, near Guadalcázar, San Luis Potosí, where it is associated with quartz, pyrite, and antimony, along the contact between granite and limestone. The product is used in the steel industry at Monterey and in the manufacture of hydrofluoric acid.<sup>26</sup>

*Argentina.*—Fluorspar occurs at San Roque, Córdoba Province, in fissure veins cutting biotite gneiss east of its contact with Andean granite. The value is doubtful on account of remoteness from markets.<sup>21</sup>

*England.*—In England fluorspar occurs in Derbyshire, Durham, Cornwall, Devon and North Wales.<sup>8,21,26</sup> The veins average 6 ft. in width and their greatest length is 7 miles. They cut Carboniferous limestone and contain also barite, calcite, galena and sphalerite. For a time the output was in excess of home requirements and much was exported to the United States. At Castleton, Derbyshire, was obtained the famous "Blue John" spar, used for ornaments. The mines originally were worked for lead, and fluorspar was rejected, but later it was produced from the waste heaps, and as these are now nearly exhausted the mineral is recovered as a by-product. In the Weardale district, Durham, fluorspar is the principal constituent of fissure veins cutting sandstone, shale and limestone, and is associated with quartz, calcite, galena and pyrite. In Cornwall and Devon the spar is associated with copper, lead, tin and tungsten minerals.

*France.*—The most valuable deposits of fluorspar in France are at Var, accessible to Mediterranean ports. In Saône-et-Loire "spar" veins cut granite-porphry and are associated with chalcedonic silica, barite, quartz and limonite. Mining and milling have developed remarkably since the World War. Steel and munitions works consume part of the output.<sup>21</sup>

*Germany.*—Fluorspar is widely distributed in Germany, reserves are considered abundant, mining and milling operations are modern and



efficient, and much of the product is exported to the United States and other countries.<sup>21,26</sup>

*Spain.*—Fluorspar is widely distributed in seven or eight districts; it is mined at a few localities with lead as by-product. Reserves appear to be ample to supply small domestic demand and some exports.<sup>21,26</sup>

*Italy.*—Fluorspar occurs in Lombardy, Trentino and Venetia Provinces. At the Rabenstein mine, at Trentino, it occurs in fissure veins associated with galena, sphalerite, quartz, calcite and broken schist. This mine, originally operated for silver-bearing galena, was reopened for fluorspar during the World War by military authorities. Both the mine and plant are said to be well equipped.<sup>21,26</sup> Some optical fluorspar is reported.

*China.*—Fluorspar mined by coolies in eastern China is exported to Japan and the United States.<sup>21</sup>

*Africa.*—Veins of fluorspar occur in South Africa and Southern Rhodesia cutting sedimentary rocks and granite, and associated with sphalerite, galena, scheelite, molybdenite and tin ores. The exports, generally of high grade, are to Australia and the United States.<sup>21,26,34</sup>

*Australia.*—In eastern Australia fluorspar occurs in veins, lodes and vugs in sedimentary rocks and granite, associated with silver-bearing galena and other minerals. The "spar," produced partly as tailings, is used locally in steelworks at Newcastle, and small quantities are exported.<sup>21,26</sup>

#### POLITICAL AND COMMERCIAL CONTROL

The importance of fluorspar in the smelting of metals, in the manufacture of steel, aluminum, Portland cement, ceramic and chemical products, optical instruments, and refrigerants, renders it of particular value in time of war as well as in peace, and raises it almost to the rank of a strategic mineral.<sup>6</sup> This was demonstrated by the Italian military authorities in undertaking its production from local mines rather than to risk having imports cut off by blockades, and by the great increase in price and production of the mineral in the United States during the World War when imports were curtailed. Fluorspar, indeed, would be accounted a strategic mineral in any large country that did not possess adequate and fairly accessible supplies. Recent Ohio River floods<sup>9</sup> illustrate how fluorspar production is cut off by high water and that the deposits might be rendered permanently unavailable if the mining area were inundated by damming that great river below the mouth of the Tennessee.

During the World War domestic deposits of fluorspar were drawn upon heavily, but they proved adequate to supply the demand. Imported spar, however, was readily sold whenever available, as it was needed at times to relieve temporary shortages caused by difficulties in min-

ing and transportation. The initiative of speeding up production of fluorspar in response to war demand was taken entirely by private interests and Government encouragement was not necessary beyond the placing of fluorspar mining in a preferred class in 1918, in order to assist the industry to hold part of the labor necessary to operate the mines.

In the summaries of domestic and foreign deposits it is shown that most of the principal countries possess supplies of fluorspar, and it is probable that many of them possess reserves sufficient for peace and war-time needs. The control of fluorspar deposits in the United States and abroad appears to be commercial rather than governmental, although, as in Italy during the World War, it is a simple matter for military or other government authorities to assume charge of production of a mineral commodity during an emergency. Foreign ownership of fluorspar deposits in the countries under review does not seem to be as significant at present as is that of certain other materials such as petroleum, iron and copper ores, and bauxite. Local ownership of fluorspar deposits still predominates in the United States, but a trend that has taken place in the last two decades is toward the acquisition of important mines of this valuable fluxing material by certain manufacturers of steel and aluminum that are large consumers of fluorspar, and also toward the contracting for the entire output of mines operated by independent producers.

TABLE 1.—*Tariffs on Fluorspar*

Effective Date	Duty	
	Long Ton	Short Ton
Prior to 1909 . . . . .	None	None
August, 1909. ....	\$3 00	\$2.68
October, 1913 .. . . .	1.50	1.34
September, 1922 . . . . .	5 60	5.00
November, 1928. . . . .	{ 5.60 (if more than 93 per cent $\text{CaF}_2$ )	5.00
	{ 8.40 (if not more than 93 per cent $\text{CaF}_2$ )	7.50
June, 1930. ....	{ 5.60 (if more than 97 per cent $\text{CaF}_2$ )	5.00
	{ 8.40 (if not more than 97 per cent $\text{CaF}_2$ )	7.50

The history of the tariff on fluorspar<sup>21</sup> may be summarized as in Table 1.

## PRODUCTION AND CONSUMPTION

An interesting outline of the history of production of fluorspar has been prepared by Hatmaker and Davis,<sup>21</sup> from which many of the following facts are derived:

Agricola (1529) considered fluorspar a highly useful fluxing agent, but its extensive use did not develop until just before the beginning of the twentieth century. Discoveries of fluorspar, or "fluat of lime," were recorded in 1814-1816 near Franklin Furnace and Hamburg, N. J., and in other eastern and New England States; the southern Illinois occurrences were reported as early as 1818, and deposits in Tennessee and Kentucky a few years later. In the West fluorspar was recorded in Colorado in the late sixties, and in New Mexico, Arizona, and other western states soon afterward. The first recorded use of American fluorspar apparently was in 1823, when 2 oz. of pure fluorspar from Shawneetown, Ill., was used, with 4 oz. of sulphuric acid, in making fluoric acid. In 1837 fluorspar, mined from a vein near Trumbull, Conn., was used with magnetic iron pyrite in the smelting of copper ores.

Mining of fluorspar in southern Illinois began near Rosiclare in 1842; shipments apparently began about 1870, in Kentucky about 1873, and in Colorado in the early seventies. The principal uses of fluorspar up to 1887 were in the manufacture of glass, enamels and hydrofluoric acid, with smaller quantities used as a flux in iron foundries and in smelting gold, silver, copper and lead. At this time the annual requirements were about 5000 short tons, but in 1888, with the commercial manufacture of basic open-hearth steel in the United States, the production of fluorspar increased to 6000 tons. With the rapid increase in the production of open-hearth steel as well as substantial increases in the use of fluorspar in the ceramics and chemical industries, and the discovery of additional uses, the demand for fluorspar likewise increased, reaching a peak production of 264,817 tons during the World War year, 1918. The total production of fluorspar in the United States from the beginning of operations to 1935, inclusive, has probably amounted to approximately 3,675,000 short tons, of which Illinois has contributed about 61 per cent, Kentucky about 32 per cent, Colorado about 5 per cent, and New Mexico most of the remaining 2 per cent.<sup>21</sup>

The domestic consumption of fluorspar is greater than production, the additional spar becoming available through the imports. Beginning with 1926, when statistics of actual consumption by various industries become available, to 1935 the annual consumption has ranged from 56,000 tons in 1932 to 196,200 tons in 1927, and has averaged 137,746 tons. The imports of fluorspar into the United States, 1915 to 1935, ranged between 6229 short tons in 1921 and 75,671 short tons in 1926,

and averaged 30,969 short tons per annum. In 1934 they were 16,705 short tons and were derived from the following countries in order of rank: Germany, 8224 short tons; Spain, 4914 tons; Union of South Africa, 1997 tons; Newfoundland, 745 tons; England, 466 tons; Canada, 187 tons; China, 112 tons; Italy, 60 tons. Germany has been the chief source of foreign supply since 1927; prior to that year England held the lead. Fig. 1 shows the curves of domestic production, imports, and average prices of fluorspar in the United States, 1915 to 1935, and the consumption from 1926 to 1935.

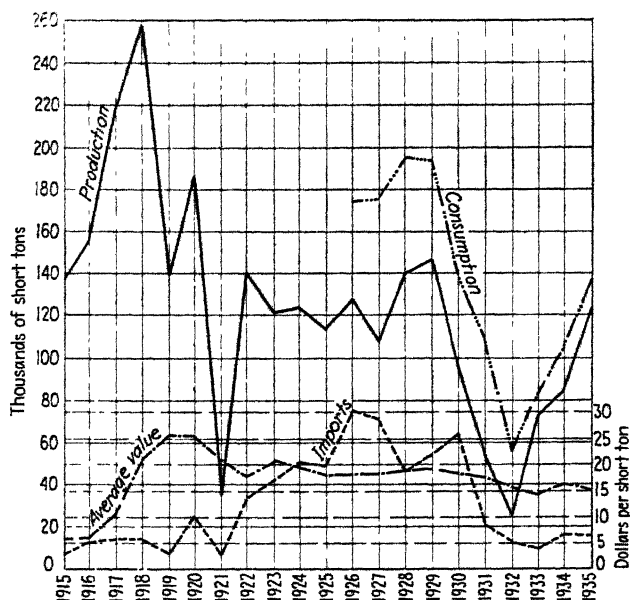


FIG. 1.—TRENDS IN PRODUCTION, AVERAGE ANNUAL VALUES PER TON, AND IMPORTS OF FLUORSPAR IN THE UNITED STATES, 1915–1935, AND OF CONSUMPTION, 1926–1935.

The relation of total production to known or estimated reserves of mineral resources is of interest. General estimates indicate that approximately 5,500,000 to 6,000,000 tons of fluorspar may still be available in the Kentucky-Illinois district and that more than 1,000,000 tons may be available in the Western States. With an average consumption of 175,000 tons per annum this total quantity would last 37 to 40 years. This estimate does not take into account the imports, which, if continued, will prolong the life of our domestic reserves, nor the possibility of discoveries of larger domestic reserves or of higher recoveries of low-grade ores through more intensive beneficiation.

Government statistics of production of fluorspar in foreign countries<sup>26</sup> are available only for the years since 1913, and are incomplete, particularly in the early years. Data for 1915, which did not include the output

for certain important countries tabulated in later years, showed that Great Britain, Spain, Norway, New South Wales, Bavaria and Saxony produced a total of 39,172 metric tons compared with 124,230 metric tons produced by the United States. In 1918, the year of greatest production in the United States, only nine producing foreign countries are recorded, then follow records for varying numbers of countries up to

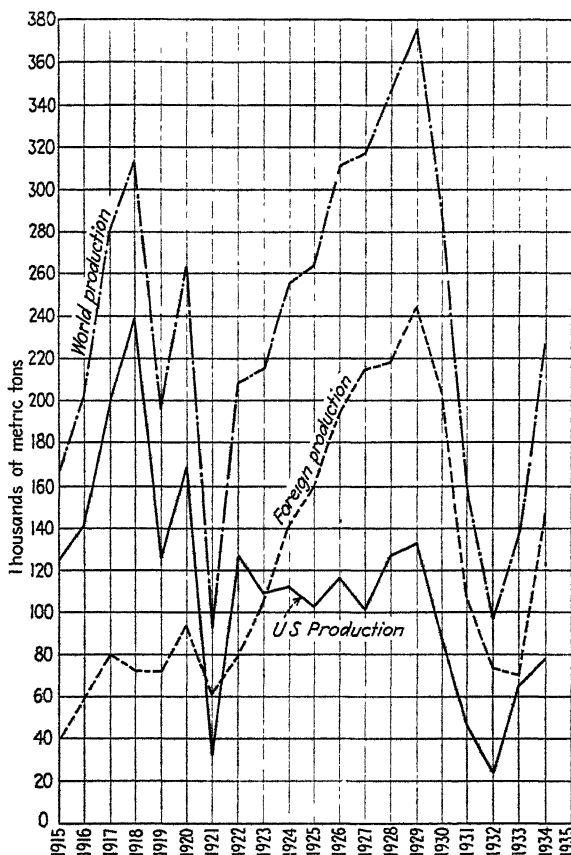


FIG. 2.—TRENDS IN PRODUCTION OF FLUORSPAR IN THE UNITED STATES, IN FOREIGN COUNTRIES, AND IN THE WORLD, 1915-1934.

as many as 20 in 1934. In 1929, the year of largest foreign production, 14 countries produced 243,383 metric tons compared with 132,847 tons produced by the United States and 376,230 tons produced in the world. From 1915 to 1934 the United States produced annually from 32 per cent to 76 per cent of the world's production and over this period produced about 48 per cent of the total. Fig. 2 shows the curves of recorded production of fluorspar in metric tons in the United States, foreign countries, and the world from 1915 to 1934. It indicates the general paral-

lelism between the curves, and also shows that foreign production exceeded that of the United States in 1921 and 1924 and has remained permanently higher ever since.

#### PROSPECTING, EXPLORATION AND MINING

Methods of prospecting and exploration for fluorspar deposits vary considerably according to the region and type of deposit concerned. Primary guides for prospecting are surface indications. In the Illinois-Kentucky district the outcrop of a vein is marked in places by the presence of gravel fluorspar in the soil or subsoil; a fault fissure is indicated by rocks of unlike character in contact along a horizontal plane and may be marked, for example, by a ridge or low bluff of sandstone that has resisted weathering more than the limestone or shale on the other side of the fault, or by a sharp difference in the soils—deep red clay soil on one side and light sandy soil on the other. In the West, where fluorspar may be an abundant gangue mineral, fluorspar float, or even fluorspar sand brought up in anthills, may indicate the presence of veins. Accurate identification of minerals is essential. A large vein of fluorspar in Colorado was not recognized for years because of its resemblance to amethystine quartz, with which the miners were familiar in a neighboring mine. In Illinois and Kentucky geophysical surveys designed to detect the presence of concealed faults have recently been made. This work, carried on in 1933 to 1935 by the Illinois State Geological Survey and the United States Geological Survey, employed electrical resistivity methods to indicate abrupt transitions between beds of different resistivity that have been brought into juxtaposition through faulting.<sup>14</sup> Checking of the resistivity surveys may be followed by trenching or churn drilling at right angles to the supposed strike of the fault.

Known fault fissures are prospected for fluorspar values in a variety of ways, most of them simple and time-honored, such as trenching, test-pitting, sinking shallow shafts, churn drilling, and diamond drilling at an angle of 45°. In exploring ore bodies in advance of mine workings, in locating faults, and in piloting rock crosscuts, underground diamond drilling, if properly interpreted, is valuable.<sup>20</sup> In prospecting for bedding deposits remote from outcrops, churn drilling is most practicable, as in much of the area that is probably ore-bearing the mineralized zone lies at depths of 100 to 250 ft. In general, a fluorspar deposit to be successfully mined must fulfill certain essential requirements: it must be favorably situated with respect to markets, transportation facilities, and water supply; it must possess a reserve of good-quality spar sufficient to produce about a carload a day for not less than 15 years; the structural relationships of the deposit must be favorable to economical mining. Some years ago (ref. 18, 1921, pp. 67–68) the cost of properly developing a fluorspar deposit and of installing adequate equipment for mining and cleaning the

ore ranged, according to the size of the deposits, from \$25,000 to more than \$1,000,000.

*Mining Methods.*—The methods employed in mining fluorspar in the United States vary considerably, according to the nature of the deposits and the technical and financial ability of their proprietors.<sup>12,23,30</sup> Many failures have been due to lack of careful consideration of the situation and undue haste to attain immediate production rather than to develop permanent mines. Fluorspar mining is in many respects similar to the mining of metalliferous deposits either in steeply dipping veins or in flat-lying lens-shaped beds, and the spar, whether it occurs as the predominant mineral or as a recoverable gangue mineral, is known as "ore." Mining is done most commonly by shafts and tunnels or "drifts," and in a small way by means of open cuts. Where the disintegrated material on the weathered outcrop of a vein contains valuable fluorspar in the form of residual gravel or lumps mined with clay and sand, this material is mined from open pits by simple hand methods or by steam shovel.

*Drift Mines.*—The bedding deposits of fluorspar at certain mines near Cave In Rock, Illinois, crop out above the general level of the surrounding country and are reached by tunnels. Where of workable thickness (usually more than 24 in.), the ore is extracted by a modified room-and-pillar method. Tunneling is also applicable to vein deposits in regions of rugged topography where the veins dip steeply and rise high above drainage. At Wagon Wheel Gap, Colo., the vein zone has been traced to a height of about 700 ft. above the creek level, up a steep slope and along a ridge of rhyolitic tuffs and breccias for a distance of more than one-half mile. At the top there is an open cut on the vein and below this four or five tunnels have been driven on the strike of the vein zone to distances varying from 400 to 1500 ft. Mining methods in use there are described by Ladoo.<sup>23</sup>

*Shaft Mines.*—Aside from prospect shafts and small, shallow mines that have not been developed adequately for efficient deeper operations, the standard practice for permanent mines on vein deposits in Illinois and Kentucky are described by Ladoo<sup>23</sup> and Reeder.<sup>30</sup> The most serious problem in the shaft mines on the Fairview-Rosiclare vein is that of handling the large quantity of water that enters the mines through channels in the limestone and from the Ohio River where it intersects the vein. The principal mines were flooded repeatedly, and by January 1924 as much as 3400 gal. per minute was being raised at the Rosiclare mine. The pumps could not stand this continuous heavy load, therefore they were stopped and there has been no further production from this part of the vein (ref. 4, p. 11).

In recent developments of bedding deposits in the Cave In Rock district, Illinois, two 160-ft. shafts have been sunk to the ore. The ore

is mined by means of an irregular room-and-pillar system, the development closely following the ore bodies, the maximum thickness of which is about 12 ft., and the average mined thickness about 4 ft.<sup>14</sup>

#### PREPARATION FOR MARKET

Crude fluor spar contains other minerals and fragments of country rock, which have to be eliminated by mechanical treatment. The methods of treatment depend upon the nature and quantity of the associated impurities, the character of the ore, and the use that is to be made of it. Some deposits contain such pure ore that hand cobbing and sorting yields marketable lump ore, but it is not practicable to carry on such preparation on a large scale. Impurities commonly associated with fluor spar in Illinois and Kentucky include calcite, quartz, clay and sand, and wall rocks such as sandstone, shale and limestone, none of which are especially harmful except to detract from the percentage of effective calcium fluoride; but they also included barite, galena, sphalerite, pyrite, and other compounds of lead, zinc, and iron, which are generally harmful. In western districts similar impurities are present as well as other metallic and non-metallic minerals and different wall rocks, such as granite, andesite and rhyolite. Some of the methods used in separation of impurities from fluor spar are: selection in the mine, crushing, washing with jets of water or in log washers, hand picking on belts, screening, gravity concentration by jigs and tables, and flotation. The processes may be very simple, such as crushing, passing through single or double log washers, and screening the spar from residual gravel deposits in order to prepare it for flux in open-hearth steelmaking; or they may be very complicated, as in preparation of vein spar for the ceramic trade or for acidmaking, where it has to be freed of practically all impurities and reduced to great fineness.

A simple, but crude, method of air separation of fluor spar from galena has been employed in Arizona. It consisted of a small table having perforations through which air was forced from below. While a good separation of the galena was effected the disadvantage was that much of the spar, in the form of fine to coarse sand, was mixed with silica and other waste. Decrepitation has been used in New Mexico and British Columbia (heating of fluor spar to about 1200° F., when it tends to fly apart and can be separated from the gangue, which is not so affected). The size and capacity of fluor spar mills vary greatly, from the small single-log washers costing a few hundred dollars to the elaborate mills in southern Illinois built to handle about 40 tons of crude ore per hour and costing at least \$500,000.<sup>23</sup>

E. C. Reeder<sup>21</sup> describes milling methods and costs at a large mill at Rosiclare, Ill., and illustrates by diagrams of flowsheets the sizing and sorting section of the plant, the tailing section and the concentration



section, and gives a plan and sectional elevation of the mill building, besides drawings of jigs. Flotation is the latest process to be applied in fluorspar concentration. It appears to have been an outgrowth of an unbalanced condition between supplies of gravel spar and of acid spar. Results of preliminary research in this field have been briefly outlined by Coghill and Greeman,<sup>11</sup> and acid and ceramic grades of concentrates are now being recovered by this process.

#### TESTS AND SPECIFICATIONS

Chemical analyses are practically the only tests required of fluorspar for use in the metallurgical, ceramic and chemical industries, but for use in optical instruments physical tests are required. Fluorspar suitable for optical purposes is found sparingly in Illinois and Kentucky.<sup>28,20</sup> The National Bureau of Standards requires that this material must be as clear as glass, free from cloudiness, inclusions, and cracks or incipient cleavage marks. Colorless material is most desirable but material faintly tinged with yellow or green may be of value. Material should be submitted from which clear pieces  $\frac{1}{2}$  to 2 in. in diameter can be cut.

There are several fairly definite grades of commercial fluorspar that are based on utilization.<sup>21</sup> In the absence of standard specifications individual consumers generally have specifications for fluorspar to suit their needs. For acid-grade fluorspar the required calcium fluoride content is usually not less than 98 per cent and the silica and calcium carbonate content not more than 1 per cent each. Barite and lead, zinc, and iron minerals are highly objectionable. This grade is sold as No. 1 lump or as ground to pass 80 to 100 mesh. The glass and enamel grade of spar is usually required to contain not less than 95 per cent calcium fluoride and not more than 3 per cent silica, 1 per cent calcium carbonate, and 0.12 per cent ferric oxide. It must be practically free of lead, zinc and sulphur. It is usually ground to 100 mesh or finer, and must be pure white. For certain kinds of glass, lower grades of fluorspar can be used.

The standard gravel form of spar used as flux in the metallurgical industry should carry not less than 85 per cent calcium fluoride, not more than 5 per cent silica, and not more than 0.3 per cent sulphur. At some steel plants, especially in the West, spar carrying as little as 80 per cent calcium fluoride and 6 to 7 per cent silica is acceptable. The gravel is usually required to pass a 1-in. screen, and fines should not constitute more than 15 per cent of the total. No. 2 lump, of various sizes up to 5 or 6 in. in diameter, is used as a flux in iron foundries for making high-grade castings.

#### MARKETING AND USES

The bulk of the fluorspar marketed in the United States, and most of that imported, is used in the steel industry, principally in the basic

open-hearth process, in which it is added as a flux to the furnace charge, to give fluidity to the slag and facilitate the passage of objectionable impurities, such as sulphur and phosphorus, into the slag. Fluorspar has been shown to possess considerably higher quantitative efficiency as a flux than limestone, especially in the smelting of refractory ores, but in all metallurgical operations the proportions of the spar that can be used are limited, since its favorable effects do not increase indefinitely as the quantity is increased.<sup>6</sup> The average quantity of fluorspar used per ton of basic open-hearth steel made by individual plants varies widely, but in general in 1932 it was between 7 and 8 lb;<sup>18</sup> its cost is, therefore, a relatively small item in the cost of manufacturing a ton of steel. The minor metallurgical uses of fluorspar or its derivatives are many and varied, including use as a flux in iron foundries, in the manufacture of alloy steels and ferroalloys by the electric-furnace process, in the production of nickel, Monel metal, and brass, in the manufacture of fluorides of iron and manganese for steel fluxing, the smelting of ores of gold, silver, copper and lead, in refining copper, lead and antimony, and in the extraction of aluminum from bauxite. In the latter process hydrofluoric acid is caused to react with aluminum hydrate or oxide and sodium carbonate to form a synthetic cryolite, to which alumina is added and from this bath metallic aluminum is extracted by electrolysis.

In the ceramics industry fluorspar is of value in the manufacture of opalescent, opaque, and colored glass, enamels, facings for bricks and Vitrolite, and in the manufacture of Portland cement. Some miscellaneous uses are as a bonding for the constituents of emery wheels and in the manufacture of carbon electrodes.

In the chemical industry fluorspar is used in the extraction of potassium from feldspar and from Portland cement flue dust; to facilitate fusion and contact of ingredients in the manufacture of calcium carbide and cyanamid; and in the manufacture of hydrofluoric acid, the key fluorine-bearing chemical, and its derivatives, the inorganic and organic fluorides, and the silicofluorides. Among these are many industrial fluorine compounds, including valuable insecticides, preservatives and dyestuffs. A new demand that is rapidly being developed for acid-grade fluorspar is in the manufacture of a refrigerating medium, known as "Freon" or "F-12." This synthetic organic compound, dichlorodifluoromethane, is reported<sup>20</sup> to be nonexplosive, noninflammable, and practically nontoxic. Other organic compounds containing fluorine that possess similar properties have been developed and in view of the expansion in the field of refrigeration and cold storage, and of air conditioning, an increasing market is promised for high-grade fluorspar.

A very small quantity of fluorspar is used for optical purposes, such as correcting the color and spherical aberration errors in lenses of spectro-

scopes, microscopes and small telescopes. Jewelry and stone ornaments have been made from colored fluorspar.

During the five years 1926-1930, the shipments of fluorspar from mines in the United States averaged as follows: according to quality—metallurgical, 83.8 per cent; ceramic, 7.5 per cent; acid, 7.3 per cent; other grades 1.4 per cent; according to size—gravel 87.1 per cent; ground, 8.3 per cent, and lump, 4.6 per cent.

An outline of marketing practice is as follows (ref. 18, 1926, pp. 35-36):

Prices of domestic fluorspar are quoted on the basis of the short ton (2,000 pounds) f o.b. cars at the nearest shipping point to mills or mines. Gravel and lump fluorspar are generally shipped in bulk in open-top cars. Ground fluorspar is shipped both in bulk in box cars lined with heavy paper and packed in bags with a capacity of 125 pounds each or barrels with a capacity of from 450 to 500 pounds each. . . .

Most of the large consumers buy the bulk of their fluorspar on contract, generally covering a definite tonnage to be delivered within a stated time and specifying the minimum content of calcium fluoride and the maximum contents of impurities that will be accepted. The contract may or may not include penalties for excesses of impurities above the specified limits. Premiums, however, are seldom paid for unusual purity.

Most of the domestic fluorspar of commerce is sold through established sales agencies, who handle other raw materials used in the iron, steel, ceramic, and chemical industries and are thus in close contact with the consumers. Such sales agencies either operate their own mines or have contracts with producers whereby the producer agrees to supply and the sales agency agrees to handle the entire output of the producer. . . .

Costs of production and transportation limit the markets in which sellers of fluorspar can profitably compete; the import duty further limits the market for imported fluorspar. The cost of producing fluorspar abroad, chiefly in England, Germany, and France, is relatively so much lower than the cost of production in the Illinois-Kentucky district that notwithstanding a duty . . . fluorspar imported from these sources is sold in western Pennsylvania and to a small extent in eastern Ohio in stiff competition with that from the Illinois-Kentucky district. The market in this area is therefore more or less divided between domestic and imported fluorspar.

#### PRICE

The most generally available data on prices of fluorspar are the average annual prices by states and grades published by the U. S. Geological Survey and U. S. Bureau of Mines,<sup>28</sup> and the quoted prices, by grades and by months, of domestic fluorspar at the mines in certain districts, and of imported spar at seaboard, duty paid, published in technical and trade journals.

Prior to the entrance of the United States into the World War, or from 1880 to 1916 inclusive, the average prices of fluorspar of all grades were comparatively uniform, ranging from about \$4.00 to \$8.21, and averaging \$6.07 per short ton. From 1917 to 1935, inclusive, the average price for all domestic spar practically trebled, rising to \$18.41, with a range of from \$10.45 in 1917 to \$25.49 in 1919. Since 1920 prices have ranged between \$14.25 and \$21.00 per ton. The sharp increase in war-time demand for fluorspar, coupled with curtailed imports and higher

production costs, caused the great advance in prices at that time; high production costs have prevailed since the war and the prices have never returned to prewar levels.

Market conditions in the Pittsburgh and Chicago districts generally set the price level for Illinois-Kentucky fluorspar. Price quotations are on domestic spar f.o.b. cars or barges at the nearest shipping point to mines or mills, or on imported spar f.o.b. ports of entry. The quoted prices by months do not always correspond closely with average annual prices. Local changes, such as bringing in new deposits mined at lower costs, large stocks, and keen competition affect price levels. Prices quoted for small lots are generally slightly higher than for large tonnages sold on contracts. The cost to consumers of foreign spar includes the foreign, or export, value, the duty, loading charges at docks, ocean freight, insurance, consular fees, and freight from docks to manufacturers' plants. In 1935 the average selling prices at tidewater, duty paid, of imported fluorspar sold to consumers were as follows: to steel manufacturers, \$18 per short ton; to manufacturers of glass and enamel, \$25.70, and for manufacturing hydrofluoric acid, \$24.60.

Imported spar has an influence on the price level at least as far west as the Pittsburgh district and has made keen competition between domestic and imported spar at plants near the Atlantic seaboard. Under normal conditions the market for metallurgical fluorspar is a buyer's market, but if changes in world conditions curtail imports, as during the World War, a seller's market ensues and the seller can dictate specifications, prices and terms of sale.

The price curve (Fig. 1) reflects in a general way the curve of production but does not show such sharp peaks or depressions.

#### CRYOLITE

Cryolite is a double fluoride of aluminum and sodium ( $3\text{NaF} \cdot \text{AlF}_3$ ), which should contain, if pure, 12.9 per cent aluminum, 32.8 per cent sodium and 54.3 per cent fluorine. It crystallizes in the monoclinic system, its hardness is 2.5, specific gravity 2.95 to 3.00, and the color of the pure mineral is snow white. Contamination with fluorite, pink quartz, chalcopyrite, pyrite, siderite, galena, sphalerite, and other minerals colors the cryolite dark in places. The mineral fuses at  $950^\circ \text{C}$ . or  $1742^\circ \text{F}$ . with practically no loss in firing. It is almost insoluble in water.

The mineral is comparatively rare, the only three occurrences commonly known being a large deposit at Ivigtut, Greenland (the only locality at which the mineral occurs in commercial quantity) and small quantities in the rocks at Pikes Peak, Colo., and at Miask, in the Urals.

At Ivigtut the cryolite is associated with pegmatite within an intrusive

mass of porphyritic granite. It lies on the shore of Arsuk Fjord conveniently situated for mining and shipping the product. The mine is worked from slopes leading from the bottom of an open quarry about 500 ft. long, 60 to 160 ft. wide and 150 ft. deep.

The mine is owned by the Danish State and the mining concession by the A/S Kryolith Mine og Handels Selskabet, Copenhagen. The raw output is divided between the Pennsylvania Salt Manufacturing Co., of Philadelphia, and the Oresunds Chemiske Fabriker, of Copenhagen.

In preparation for marketing, the ore is first screened, then hand-picked, crushed to pass  $\frac{1}{4}$ -in. screen, passed on continuous rubber belts under high-power magnets and separated into two sizes, the coarser of which is treated in jigs and the finer over Wilfley tables and jigs. After drying and further magnetic treatment, the two sizes are mixed and ground.

Three grades of refined cryolite are marketed. The highest contains a minimum of 98 per cent sodium aluminum fluoride (cryolite) and a maximum of 1.5 per cent silica and 0.25 per cent iron oxide, with lime seldom more than 0.1 per cent. The next two grades, which differ mainly in fineness, carry 93 to 94 per cent sodium aluminum fluoride and not more than 0.75 per cent iron.

Cryolite is used as a metallurgical flux and solvent largely in the manufacture of aluminum, but here it is meeting competition in the form of artificial cryolite, which is made from sodium carbonate, bauxite and fluorspar, or from their derivatives, which supply sodium, aluminum and fluorine. Other uses are in the manufacture of enamels, opaque glass, glazes, binder for abrasives, electric insulating material and insecticides.

The production of cryolite in Greenland in 1936 amounted to 17,135 long tons compared with 23,100 tons in 1935 and 14,999 tons in 1934, with a large increase predicted for 1937. Available statistics concerning cryolite, including artificial cryolite, are those of imports for consumption in the United States. In the period 1915 to 1935, inclusive, the imports ranged from 1950 long tons valued at \$97,500 in 1918 to 8315 long tons valued at \$695,794 in 1930, and the average annual price per ton ranged from \$21 in 1915 to \$36.25 in 1929. In 1916 the average price apparently increased to \$42.84 per ton, because of the dangers and difficulties of ocean shipping during the World War, and, like prices of fluorspar, they have still further increased and remained high ever since. The total imports for consumption, 1915 to 1935, amounted to 114,265 long tons, valued at \$7,745,216, which represents an average annual quantity of 5441 tons and an average annual price of \$67.78 per ton. Cryolite is imported free of duty.

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## CHAPTER XVI

### PRECIOUS STONES

BY SYDNEY H. BALL, MEMBER A.I.M.E.

**M**INERALS used primarily for personal adornment and decorative purposes are called precious stones. To be so prized, the stones must possess beauty of color, must not be too common, and must be hard enough to withstand ordinary wear. If transparent, they must have brilliancy and fire. Almost one hundred minerals have been used as precious stones, the noble gems being the diamond, emerald, ruby, and sapphire; pearls are frequently included by courtesy. These gem stones, however, are sometimes equaled in beauty by fine opals, aquamarines, tourmalines, spinels, chrysoberyls (both cat's-eye and alexandrite), and spodumenes (hiddenite and kunzite).

The Greeks, Romans, and medieval Europeans grouped together gem stones according to color and degree of transparency: and only as the niceties of mineralogy began to be known, about a century ago, was it realized how many minerals have the beauty required of gem stones. Pliny, for example, did not distinguish between rubies and the red varieties of spinel and garnet, although he conjectured that the beryl and emerald were one mineral.

#### GENERAL SURVEY

Early man treasured bright colored berries, attractive shells, white teeth of wild animals and brightly colored pebbles, and used them as personal ornaments.

In the eastern hemisphere, the earliest stones used were members of the quartz family (100,000 to 75,000 B.C.): then obsidian and amber (50,000 to 25,000 B.C.): the jade minerals, fluorspar and jet (22,000 to 7,000 B.C.): turquoise (7000 to 3400 B.C.): lapis lazuli and garnet (prior to 3500 B.C.): emerald (2000 to 1800 B.C.): sapphire and ruby (600 to 500 B.C.) and diamond (480 B.C.). Indeed, gem mining is the oldest form of mining, and before any metals were known primitive man recognized about eighteen gems and decorative stones. At first he sought them in stream gravels and residual deposits but by 3400 B.C. the turquoise mines of the Sinai Peninsula were operated. This was the world's first important hard-rock mining enterprise. The Afghanistan



lapis lazuli mines may be almost as old. The emerald mines of the Egyptian Red Sea coast were operated at least as early as 1925 B.C., and the olivine mine of Zebirget Island, Red Sea, was opened between 1580 to 1350 B.C. It is believed that the diamond deposits of India and the sapphire-ruby mines of Ceylon were producing as early as 800 to 600 B.C. The mines of amber in Prussia were the most important from some time prior to 7000 B.C.: the center of production then moved to the borders of the Red Sea, from which in about 200 B.C. it was transferred to India and Ceylon. First place was usurped in 1725 A.D. by Brazil, which in turn was superseded by South Africa in 1870. Similarly, the center of the world's trade in gems has been successively Babylon, Alexandria, Rome, Venice, Amsterdam, and now London, Paris, Antwerp, Amsterdam, Bombay and New York.

When the white man first appeared on our shores the American Indian was using about as many precious and decorative stones as were the people of the eastern hemisphere. He depended mainly on gravels for his gems but his underground operations for turquoise and obsidian were large. He also mined emerald, quartz, and many decorative stones. The trade in gems was wide; that in New Mexican turquoise extended from Yucatan to Wisconsin, and the stocks of Colombian emeralds in the hands of the Peruvian Indians were so large that for at least 200 years after the Conquest the gem was known as "Peruvian emerald." The Indian used jasper and chalcedony as early as 15,000 B.C., and agate, jade, jet, turquoise and a host of other minerals were used before the beginning of our era. Amber, emerald, garnet, lapis lazuli, and opal were mined before 1000 A.D.

#### USES

By far the largest part of the world's gem production is used for decorative purposes, mainly in jewelry but to a lesser extent as material for statuettes, vases, and other *objets d'art*. For the latter, the less valuable gem stones are usually employed, although statuettes of sapphire and ruby are known. The lapidaries of China, Delhi (India) and the Urals are particularly clever in this branch of the art.

Some 15 per cent of the world's gem production by value (about 50 per cent by weight) is used in industry. The diamond is the most important of the gems industrially, its value depending upon its hardness, which is greater than that of any other substance, natural or artificial. There are three types of industrial diamonds: (1) bort, a trade name for diamonds too badly flawed or too off-color to be used in jewelry; (2) carbonado or black diamond, a closely knit aggregate of excessively small diamond crystals; and, (3) ballas, a globular mass of diamond crystals, radiating from a common center. Bort has the virtue of hardness and comparative cheapness: carbonado of extreme toughness, and ballas of

hardness and toughness. Bort is a by-product of all diamond mining and makes up about 50 per cent of a normal year's production. Carbonado comes from Bahia, Brazil, and ballas from Brazil and certain of the South African mines, notably New Jagersfontein and some alluvial deposits. Bort is relatively abundant, about 3,500,000 carats\* being produced annually. Bahia now produces from 20,000 to 30,000 carats of carbonado and ballas is still less common. Carbonado occurs in pebbles of larger average size than the gem stone, although the largest ever found (3078 carats) only approached in size the famous Cullinan diamond. Bort of good quality but small size retails for about \$7.50 per carat: carbonado is from five to ten times as expensive, and ballas may cost twice as much as carbonado. Some five years ago the price was forced to exorbitant heights (\$175 a carat) and industrialists substituted bort for carbonado for many purposes, so that the latter has permanently lost many markets. Of the multitudinous uses of industrial diamonds, the more important are: in diamond-set tools for truing emery wheels, shaping automobile and airplane parts, etc.; diamond-drilling (black diamonds were formerly used, but now largely bort, resulting in a 50 per cent reduction in costs); diamond dies, for drawing all finer wires and all those of the harder metals; glaziers' diamonds; as dust for various abrasive purposes (notably bonded in Bakelite wheels); and in the cutting of diamonds and other precious stones.

Ruby and sapphire were used for the bearings of watches, meters, and scientific instruments, but recently the synthetic corundums have largely supplanted the natural gems. Ruby, sapphire, garnet and quartz are used as abrasives. Rock crystal, Iceland spar, and tourmaline are used in various optical instruments and fused quartz for chemical apparatus. Agate mortars and pestles and agate bearings for balances are found in every chemical laboratory. Beryl is the principal ore of beryllium, and phenacite would be a valuable ore if found in quantity. Zircon is one of the principal ores of zirconium. Lapis lazuli was formerly the base of ultramarine, although now most of the color is artificially produced.

Precious stones are one of the most valuable of commodities and over a period of years their price trend has been upward. Great wealth is concentrated in them in small compass. They also enjoy an international market. In the Orient and particularly in India where bank stocks and bonds are unknown to the majority of people, precious stones have been a popular form of investment for centuries. In consequence, the family fortunes of the astute Hindus have from generation to generation enjoyed a reasonable growth. In the West, gems assume the role of concentrated

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\* For centuries the carat has been the unit by which most gems are weighed. At one time each gem center had its own carat, but now it is practically universally equivalent to 0.2 grams, or 3.086 grains (155.54 carats per troy ounce).

wealth readily hidden or easily transported when people doubt the stability of their country's currency or when political upheavals or religious persecution cause them to leave their homeland overnight.

Precious stones have been a potent factor in furthering exploration. In the Middle Ages, much of Europe's knowledge of the Eastern countries was due to the travels of gem merchants. It was the precious stones of Cipangu (Japan), together with its wealth in precious metals, that lured Columbus to explore for a westward route. In the early exploration of America, similar lure led Europeans forward, although Colombia alone with its emeralds adequately fulfilled their hopes.

#### GEOGRAPHIC DISTRIBUTION

Precious stones are widely distributed over the world, and while important commercial deposits are few, Africa, Asia, South America and Europe all have deposits of the first rank, North America and Australia alone being without them. The south half of Africa, because of its pre-eminence as a diamond producer, is the most important factor in the industry. Burma, Siam, Ceylon, the Urals, Madagascar, Brazil and southern California are notable for the wide variety of precious stones they produce.

The United States in comparison to its wealth in most other mineral resources is poor in precious stones, having no deposit of the first rank. The sapphire deposits of Montana, the turquoise mines of the Southwest and the tourmaline mines of Maine and southern California are of the second rank. California also produces kunzite. The Arkansas diamond deposit has not yet been proved to be of commercial importance. Precious stones occur near many resorts, but the tourist purchaser of the "local" product is more than likely to become the possessor of a South American stone cut in Germany and mounted in Providence, R. I. Probably no other country, however, has produced such a wide variety of precious stones as the United States, more than 60 distinct species having been produced commercially. Adequate statistics exist only from 1880 to 1924, but during those 45 years about \$9,800,000 worth of gems was produced, the peak year being 1909 (\$534,000) and the year of least production, 1923 (\$60,000). These figures cover the most active period of gem mining in the United States. Including estimates of production from 1925 to the present, the total value of the precious stones produced in the United States since 1880 has been about \$10,500,000. Montana has produced more than any other state, accounting for about 48 per cent of the total value. California and Nevada follow with 23 and 8 per cent, respectively. From 1883 to 1921 the United States produced sapphire (including a little ruby) to the value of \$3,018,406; turquoise and matrix, \$2,088,262; quartz gems, \$1,894,289; tourmaline, \$822,955; garnet, \$173,979; beryl, \$138,186; and variscite, \$125,718.

While the Mount Mica tourmaline locality in Maine has been worked in a desultory manner since its discovery in 1820, the first American gem-mining company was the Emerald and Hiddenite Mining Co. (incorporated April 1, 1882), which exploited the emerald and hiddenite deposit at Stony Point, N. C. and operated it from August 1880 to 1888. Several other companies have been formed to mine emeralds in North Carolina. In the nineties of the last century and early in the present century, supplementing the desultory mining of the Pueblo and Navajo Indians, the white man carried on rather extensive mining operations at a number of the turquoise mines of the Southwest, notably at Los Cerillos and in the Burro Mountains, New Mexico. Some of the material recovered was fine, and in certain years the production was appreciable. Should turquoise regain popularity, doubtless the mines will be reopened. The agatized wood of Arizona 50 years ago was cut and polished on a considerable scale: the fad threatened to destroy the beauty of the Petrified Forest but happily the menace disappeared when the area was made a National Park.

Sapphires were first found in Montana by placer miners in their sluice boxes along the Missouri River, in 1865. In the early eighties of the last century several thousand dollars worth was yearly produced as a by-product of placer mining. In the nineties, the gem was found in a basic igneous dike and in 1896 an English company began mining it. The work was suspended only in 1929 and in some of the intervening years the production reached \$450,000. Some very attractive, brilliant, though rather pale gems were recovered. These were sold in London and to a minor extent in Paris and New York, but 85 per cent of the production by weight was shipped to Switzerland to be cut into jewels for watches. In 1906, diamonds were found near Murfreesboro, Arkansas, in a rock very similar to the kimberlite of the South African pipes. Several companies were formed but so far the production has been only about 10,000 carats and for the past three years no company has operated.

Mesa Grande, San Diego County, Calif., has produced much beautiful tourmaline of various colors. The pink has been exported in quantity to China for cutting. Kunzite, the transparent, lilac-colored spodumene, is obtained from the same district. The Virgin Valley, Nevada, has produced some fine black opals and Latah County, Idaho, some attractive white ones. The moss agates of Montana and of Wyoming are as fine as any in the world. A number of other precious stones of American origin come on the market from time to time, notably aquamarine and golden beryl, quartz gems and agates, fine amazonstone, and splendid malachite and azurite. The ornamental use of gold quartz began in California over eighty years ago. The deep blue benitoite (California), hiddenite (North Carolina), the yellowish green variscite (Utah) and the rose red rhodolite (North Carolina) are found only in the United States.

## GEOLOGIC OCCURRENCE

Gems occur in practically all types of rocks, in gravel, in veins, and in deposits formed by cold circulating waters and those of descending oxidizing waters. Fossil vegetable matter is represented by amber and jet, and fossil animal matter by bone turquoise.

Because of the preponderant position in former times of the South African kimberlite pipes as diamond producers, the basic igneous intrusive rocks were from 1878 to 1921 the dominant original source of gem stones. Sapphire and pyrope also occur in such rocks and if the diamond market booms they may regain the first place. Potash-rich or soda-lithia-rich pegmatites are the source of a bewildering array of beautiful gems, although their combined value has been but one per cent of that of the gems obtained from the basic igneous intrusives. Burmese jadeite occurs as one of a series of soda-rich intrusives. Basic lava flows, granites and andesites furnish a few gems. The metamorphic rocks are relatively unimportant but of them contact-metamorphosed limestone (lapis lazuli and ruby) is the most important and regionally metamorphosed schists and gneisses (nephrite, etc.) come next. Opals are usually deposited by volcanic waters from acid flow rocks and agate by volcanic waters from basic flow rocks, the former in point of annual value being more important. Amethyst, rock crystal and gold quartz are the principal gems in veins of magmatic origin, and turquoise is the principal gem of supergene origin.

Most gems are heavy, physically resistant and chemically inert, and in consequence practically all gems occur in stream gravels. Indeed, prior to the discovery of the South African kimberlite pipes, stream gravels were responsible for 95 per cent of the world's gems and owing to the small diamond production from the pipes in 1935 the percentage was almost as great. Many gravel deposits through concentration and reconcentration of their precious-mineral content are profitable to work, although the source rock was relatively poor in gems. Even the excessively hard diamond may have been subjected to such long erosion that it is well rounded. Fractured and imperfect stones cannot endure nature's long milling and in consequence stream gems on the average are of better quality than mine stones. Desert gravels (some diamonds and olivines) are much more important than beach gravels. Amber and jet occur in relatively young sandstones, and conglomerates in places contain gems.

Rock weathering is important in gem mining, largely because it lessens mining costs and the danger of injuring the gem in removing it. In the weathered zone, the worthless satellites may be dissolved out or removed by wind or water. The weathered "yellow" ground of the South African mines contained in instances six or seven times the diamond content of the fresh pipe rock in depth. Many a gem mine in pegmatite is profitable only in the weathered zone.

## POLITICAL AND COMMERCIAL CONTROL

The British Empire, largely because of the diamond production of South Africa, Southwest Africa, the Gold Coast and Sierra Leone, accounts for from two-thirds to four-fifths of the world's gem production. The Belgian Congo and Angola, because of their diamond production, account respectively for about one-fifth and one-tenth, Brazil produces about 3 per cent. The minor producers, each accounting for less than 1 per cent of the total, rank as follows: Germany (amber), Siam (sapphire, ruby and spinel), Colombia (emerald), Russia (a wide assortment of gems) and Persia (turquoise). Most of the gem fields are nationally owned but Americans are heavily interested in certain of the diamond mines of the Belgian Congo and in Diamang in Angola, while Newmont Mining Corporation indirectly is heavily interested in the shares of South African diamond mines. South African pipe mines have always been favorites on the Paris Bourse.

## PRODUCTION, CONSUMPTION AND MARKETING

Precious stones are indestructible and practically all of the gems that have ever been mined exist today. Many an Egyptian or a Roman gem is worn today by an American woman or a Wall Street tycoon. Of course, in cutting all jewels are reduced in weight, which for diamonds amounts to about half that of the rough stone. Some gems have been

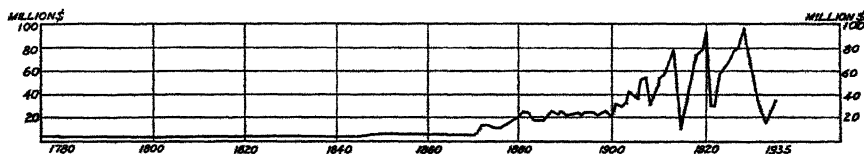


FIG. 1.—WORLD'S GEM-STONE PRODUCTION, 1780-1935.

lost in shipwrecks, a few have been beaten to powder in a fit of anger (i.e., the Pigott diamond by Ali Pasha), and many have been buried, but enterprising archaeologists recover some of the latter and permit us to study them. Gems used industrially are largely destroyed. I have elsewhere estimated that the world's stock of gems, largely in India, the United States and England, is worth nearly \$6,000,000,000.

The graph of gem production from 1780 (Fig. 1) portrays the remarkable growth of the industry, and indeed that of gem consumption, for the two are practically equivalent. In the eighteenth century the production was stabilized at from \$3,500,000 to \$5,000,000 a year; it then gradually rose to \$8,000,000 by 1871, after which the increased output of the African diamond mines caused a precipitate rise to a peak production of \$97,000,000 in 1928, with deep canyons signaling the World War, the 1921-1922 postwar depression, and the recent depression. At present a normal year's production is about 22 times that of

1700, nine times that of 1850, and three or four times that of 1900. The world's diamond mines now produce in a day as many carats as did those of 1700 in a month, but rapid as has been the growth of the industry, its expansion is relatively small compared to the increase of the production of gold and of the base metals. In 1700, diamonds accounted for about 50 per cent of the yearly production of gems, but the popularity of that gem has so increased that it now represents about 95 per cent of the total. The increase noted above is therefore largely one in diamond output.

It seems probable that alluvial mines were worked by Paleolithic man, and the production from alluvial mines was predominant to 1878 A.D., although from 3500 to 600 B.C. there were doubtless years in which the production of the Sinai turquoise, Afghanistan lapis lazuli and Egyptian emerald mines exceeded in value the production of gem placers. From 1878 to 1921 the large diamond output of the South African pipe mines caused underground production to surpass that of gem placers. A series of remarkable alluvial diamond discoveries reversed the picture and in 1930, the last year of normal diamond production, placers accounted for 57 per cent of the world's production and underground mines 43 per cent.

The United States is largely dependent upon imports for its precious stones, but the declared value of such imports varies widely with the prosperity of the country. Comparison of the imports of the boom year, 1929, and the year 1935 is given in Table 1.

TABLE 1.—*Imports of Diamonds and Other Precious Stones into the United States, with 1935 Duties*

	1929	1935
Diamonds, glaziers' and engravers', unset, and miners', free.	\$ 4,060,577	\$ 4,293,611
Diamonds, uncut, free . . . . .	9,885,072	4,261,921
Diamonds, cut but not set, dutiable, 10 per cent . . . . .	42,009,583	15,538,902
Pearls and parts of, not strung or set, dutiable, 10 per cent . . . . .	10,389,458	652,219
Other precious stones, uncut, free. . . . .	352,053	50,444
Other precious and semiprecious stones, cut, but not set, dutiable, 10 per cent. . . . .	9,007,783	1,282,348
Imitation precious stones, except opaque, dutiable, 20 per cent. . . . .		1,480,937
Imitation precious and semiprecious stones, opaque, dutiable, 60 per cent, including imitation pearls, dutiable, 40-90 per cent. . . . .	3,945,066	30,032
Marcasites, dutiable, 20 per cent. . . . .		21,976
	\$79,649,592	\$27,612,390

It is to be noted that the imports of industrial diamonds in 1935 exceeded those of 1929, although the other figures are much lower, and

this trend is expected to continue, because of the diversified uses of the mineral in industry. Belgium and Holland export virtually all of the cut diamonds, and these countries, together with South Africa and the United Kingdom, the greater part of the uncut diamonds.

Being a luxury, precious stones are normally subject to heavy duties, both in the country of their production and that of their consumption. South African diamonds, for instance, pay a 10 per cent export tax and up to a short time ago gems entering Japan paid a 100 per cent import tax. High tariffs foster smuggling, therefore in many countries there is a tendency to reduce import duties on gems.

The Diamond Corporation, through its subsidiary, the Diamond Trading Co., sells from 90 to 95 per cent of the world's diamonds, and the principal diamond-mining companies are either shareholders in the corporation or have a contract to sell their product to it. The rough diamonds are sent from the various mines by registered parcel post, the companies carrying a floating insurance policy. The rough stones are sorted in the Corporation's offices, 8 Charterhouse Street, London. In good years it disposes of them to brokers, and in periods of overproduction and subnormal demand it builds up its stock. In a normal year sales amount to \$70,000,000 to \$90,000,000.

Practically all gems are sold by the metric carat of 200 milligrams, although in certain fields local units of weight are still used. A brilliant-cut diamond,  $\frac{1}{5}$  in. in diameter, weighs about one carat, and one  $\frac{1}{2}$  in. in diameter, 9 carats. Because of its lower specific gravity, an emerald of these dimensions would weigh less.

Precious stones have ever been among the most precious of commodities, the noble gems being surpassed in value only by radium and a few other very rare elements. Their value is so concentrated that one could conceal about his person \$10,000,000 worth of fine gems. The value of a precious stone is determined by three main natural characteristics—its beauty (either fire, brilliancy, or color), durability and rarity. A fourth, artificial, is the perfection of its cutting, or, in trade terms, its "make." Less important are: an adequate supply, portability, international market, tariffs, and world economic conditions. These factors determine the value of the noble gems, for which the demand is always relatively steady, and the sales of which are in direct ratio to the world's purchasing power. Fashion, superstition, royal sponsorship, danger of competitive imitations, nationalism and effective publicity more particularly influence the price of the less valuable gems.

The world has been relatively consistent in its ranking of gems for some 1900 years, for the Romans of Pliny's time valued the diamond first, then the pearl, next the emerald, and finally the opal. Pliny does not give the rating of the ruby and sapphire. The Maharatnani, or the Five Great Gems of the Hindus, for centuries have been the diamond,



pearl, ruby, emerald and sapphire. The Persians in the thirteenth century valued the gems as follows: pearl, ruby, emerald, chrysolite and diamond. The cutting of that day did not, of course, bring out the full beauty of the diamond.

Fig. 2 is a graph of the price for the past 75 years of the diamond, emerald, ruby and sapphire, the relative sales volume of the four gems

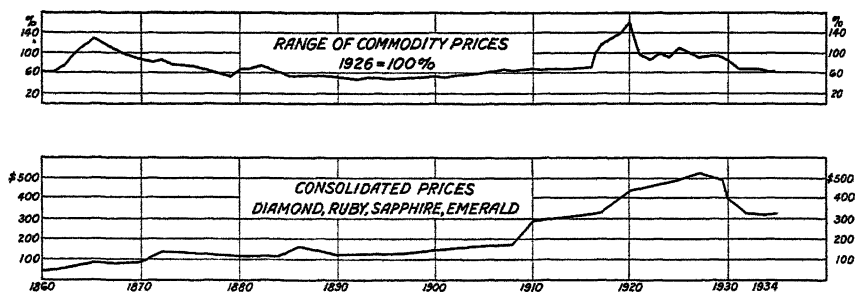


FIG. 2.—PRICES 1860 TO 1936. CONSOLIDATED DIAMOND, RUBY, SAPPHIRE AND EMERALD, WITH COMMODITY PRICES.  
(Ball: *Econ. Geol.*, 1935, 30, 638.)

not being weighted. For comparative purposes, a commodity price curve is also presented. Figs. 3 and 4 are price graphs of a one-carat, well cut, rather fine diamond, and of the emerald, sapphire, and ruby from 1778 to date. From about 26 A.D. to 1872 A.D., a one-carat white

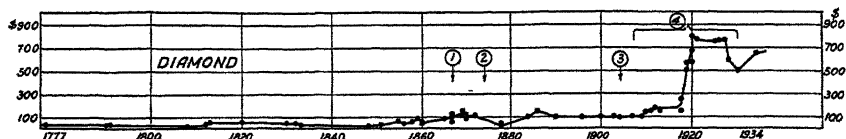


FIG. 3.—PRICE OF ONE-CARAT CUT DIAMOND, 1777–1935.

- (1) 1867, South African fields found.
- (2) 1874, importance South African fields realized.
- (3) 1904, Premier becomes a large producer.
- (4) 1907, Belgian Congo fields found; 1908, Southwest African field found; 1919, Gold Coast fields found; 1930, Sierra Leone fields found.

diamond was the most expensive stone, except from 1501 to about 1800, when the ruby usurped the lead. From 1872 to the present day the emerald has been the most expensive stone.

Exceptionally fine gems have no fixed price, and, as with fine paintings, set rules do not hold. Such are deep-colored “fancies” (red, green or blue diamonds), white diamonds of unusual size and water, rubies of over 4 carats, particularly fine emeralds, and unusually fine sapphires. About 1592, Linschoten, the famous Dutch traveler in the Orient, formulated a rule for the valuation of gems; namely, to find the value of a stone of more than one carat, multiply the square of its weight by the value of a one-carat stone of the same quality. This rule still is approximately

correct for rubies and emeralds but for the past 60 years, because of the abundance of large diamonds found in South Africa, it has been discarded in diamond valuation. Owing to the relative commonness of large sapphires, it gives too high a price if applied to that gem. The prices of the other gems are less stable; thus, for example, in the Middle Ages fine amethysts and opals were comparable in price to diamonds. At present, after the four noble gems, the other precious stones rank about as follows:

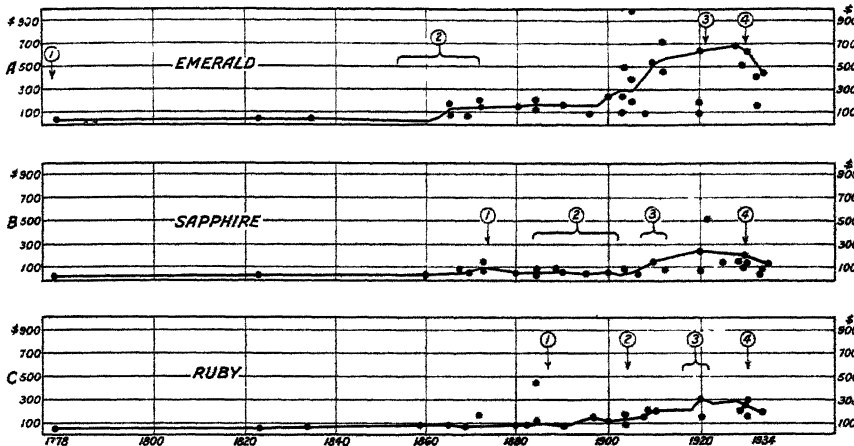


FIG. 4.—PRICES OF ONE-CARAT CUT EMERALD, SAPPHIRE, RUBY, 1778-1934. (DOTS REPRESENT REPORTED PRICES.)

Emerald: (1) 1567-1800, market depressed by oversupply of Colombian emeralds; (2) 1852-1871, very popular under Third Empire (France); (3) Stone for Princess Mary's engagement ring, emerald; (4) 1930, American tariff reduced from 20 to 10 per cent ad valorem, emphasizing effect of depression.

Sapphire: (1) 1871-1872, after Franco-Prussian War, became very popular; (2) 1883-1903, four new fields increased supply greatly; (3) 1909-1910, synthetic sapphires appeared on market; by 1912 adverse effect ended; (4) 1930, American tariff reduced from 20 to 10 per cent ad valorem, emphasizing effect of depression.

Ruby: (1) 1887, Burma Ruby Mines Ltd. started operations; (2) 1904-1905, synthetic rubies appeared on market; (3) 1916-22, ruby temporarily out of vogue; (4) American tariff reduced from 20 to 10 per cent ad valorem, emphasizing effect of depression.

(Ball: *Econ. Geol.*, 1935, 30, 629.)

alexandrite, star ruby, black opal, cat's-eye, star sapphire, demantoid, spinel, Brazilian topaz, aquamarine and pink beryl, zircon, tourmaline, white opal and peridot. Amethyst, turquoise and garnet are worth from 25¢ to \$2.50 per carat cut, while most of the precious stones not mentioned are worth still less, and in some jewelry the value is in the workmanship rather than in the material. It should, however, be added, in fairness to precious stones, that 25¢ a carat is equivalent to \$567 per avoirdupois pound (\$1250 per kilo).

#### EXPLORATION, MINING AND MILLING

In normal years, gem mining employs some 150,000 men, of whom one-fifth are whites and the rest African blacks, or other races of color.

In describing gem mining and milling, one must differentiate between the operations of the native miner and that of the companies. The former is primitive and has changed little in centuries; the latter are well conducted mining operations which make progress yearly. The native, as a rule, dispenses with prospecting, although he may locate ruby-bearing or sapphire-bearing gravel by forcing a soft iron bar into the ground and noting whether the iron is scratched. Usually he finds his deposit by the trial and error method, and if a shaft that he believes to be well located proves unprofitable, he is sure that the gods or the spirits of the mine are unwilling to disgorge its wealth.

Kimberlite pipes in South Africa are common, but only a small percentage of them contain a profitable diamond content. Geophysical methods of prospecting can outline such pipes, but such surveys are of little value, as they can indicate nothing as to grade. The presence of alluvial diamond deposits near a pipe may be encouraging but large-scale washing tests alone can prove whether development of a pipe is warranted. The large companies mining alluvial diamonds block out their gravel by systematic prospecting before beginning mining. This may consist of test-pitting on 10-m. or 100-ft. squares, careful note being taken of the thickness of the overburden and of the pay gravel, and of the nature of the bedrock. The gravel samples washed are large. In certain stream valleys, trenches across the entire valley at distances of, say, 100 ft. are more convenient. Drilling may be used in water-logged ground, although the sample recovered is too small to be of much value, except in such fields as that of the Kasai Basin (Belgian Congo), where the diamond content is proportional to the percentage of certain of the heavier and more common "satellites" accompanying the diamond.

The native producer usually obtains his gravel by surface digging, or if the overburden is thick, by sinking a pit to the gravel horizon and then extracting as much of the surrounding gravel as he thinks safe. The process is repeated as necessity requires. Timber is nonexistent or consists of simple shoring with small timbers. Hoisting is usually done in baskets and drainage by a kerosene tin on a sweep. In the Afghanistan lapis lazuli, the Burmese jade mines, and in certain of the Indian diamond mines fire setting is employed. Boulders or gravel in the larger stream beds are, in instances, recovered by divers, who descend on a rock-weighted rope.

The South African pipe mines were started as open cuts, but most of them are now underground mines, the depth of economic and safe open-cut mining having been exceeded (Fig. 2). The Premier mine (closed since 1932) is a vast oval open cut about  $\frac{1}{2}$  mile long,  $\frac{1}{3}$  mile wide, and 660 ft. deep, marked out in benches 50 ft. high. The "blue ground" is drilled and blasted, and blacks load the ore cars, hand labor here as in many gem mines being cheaper than mechanical methods. When operated at capacity, Premier was one of the world's large tonnage mining

operations. In the underground mines, a shaft is sunk in the country rock at a safe distance from the pipe and main haulageways are driven to the pipe at stated vertical intervals. From the haulageway, drifts are driven through the pipe 40 ft. apart, and smaller drifts cross these at right angles. The blue ground is caved up to the next level, retreating from the far side of the pipe. At a certain distance from the far wall, a series of steplike levels is formed. The blue ground is trammed to the shaft, dumped into ore pockets and hoisted.

The important alluvial diamond-mining companies, according to local conditions, remove overburden by sluicing, by hand or by mechanical shovels. The gravel of Southwest Africa is excavated by 5-cu. ft. electrically operated, caterpillar-mounted shovels and power shovels are used at the Namaqualand State mines; that of Angola, most of the Belgian Congo\* and the Gold Coast by pick and shovel work.

The simplest milling is that of the Indian diamond miner, who sizes his ore with a sledgehammer, washes out the mud and fines, spreads the remainder on a flat surface and picks and repicks it for diamonds. Tavernier, however, at the Kollūr mine, found that the finer materials were winnowed dry several times in a basket and then hand-sorted. The Singhalese gem hunter concentrates his gravel in a near-by stream in a rattan basket and then hand-sorts the concentrate. The Borneon diamond miners sit at either end of a trough filled with water and clayey gravel, forcing the mass backwards and forwards with their feet. The larger pebbles are thrown away. The gravel is sized with a sieve and concentrated in native pans and the concentrate is hand-sorted. The British Guiana "pork-knocker," as the local colored prospector is called, adds water to his gravel and hoes it through an iron plate with  $\frac{5}{8}$ -in. holes. The oversize is thrown away and the rest is concentrated on a sieve with  $\frac{1}{16}$ -in. holes. The Brazilian native worker places his gravel in a three-sided compartment sloping toward the stream. He then throws water on the gravel, to remove the lighter particles, concentrates the remainder in a batea, and hand-picks the concentrates.

In South Africa the blue ground from the pipes was formerly "farmed" for from 6 to 18 months, by the end of which period most of the rock had weathered to a clay. Now, however, it is crushed in stages, corrugated rolls being used. The crushed rock passes to pans 14 ft. in diameter, with rotating arms fitted with knifelike blades. The heavier particles, including the diamonds, fall to the bottom of the pan, where they are forced to the perimeter and periodically drawn off. The lighter material flows over a weir at the central core. The pan concentrates, about 2 per cent of the "blue ground," are sized and then jigged. These jig products were formerly hand-picked, mainly by native criminals, but

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\* In one instance an electrically operated shovel is used.

now are fed to a grease table, which consists of a steplike series of bronze, corrugated plates sloping gently, covered with a coating of grease. The table has a lateral, oscillating motion. Water and the concentrates are fed at the head of the table and a treatment and re-treatment results in an almost perfect separation of the diamonds, with a little pyrite, ilmenite, ruby and sapphire as by-products. The grease, together with the embedded stones, is then scraped off the table and placed in a wire basket, which is immersed in boiling water. The concentrate is treated with acid and alkalis and the diamonds screened to size and sorted by hand. A shipment is divided into about ten classes as to shape and purity and then the more valuable divisions are further classified into eight colors. Each subdivision is placed in papers, packed in boxes, carefully sealed, and sent to London by registered mail. At the Diamond Corporation's offices the packages are re-sorted, a large shipment sometimes being divided into over one thousand subdivisions.

In the large alluvial diamond mills, those of the Belgian Congo, for example, the gravel, if very clayey, is first puddled, then sized by trommels, the undersize being rejected and the oversize sent to the dump. Occasionally it passes over picking belts en route. The intermediate product is concentrated in pans from 5 to 14 ft. in diameter and the concentrate is jigged, the Harz type of jig being common. In certain instances magnetic separators remove magnetite and other magnetic "satellites." The concentrate is then delivered to containers, which are locked and trucked to a central picking station. This concentrate in the Congo mills is about 0.10 per cent of the feed.

Superintendents of operating gem properties must be much more vigilant than those of gold mines to guard against "high grading." Therefore in the more modern mills the later milling units are locked and access to them is permitted to only the most trusted employees. While the chance of a diamond being seen as blue ground or gravel is being mined is normally small, all black employees of the South African pipe mines spend the term of their enlistment (three or six months) in compounds. Here they are well housed but are effectively separated from the outside world by wire fences. When their term is about to be finished they are purged and X-rayed to be sure they have not swallowed diamonds or concealed them about their person. The natives employed in the sorting plant of the Burma Ruby Mines Ltd. were compelled to wear boxes over their heads with gauze windows, so that they could not swallow the gems. At best, however, thefts occur, in direct ratio to the primitiveness of the mining methods.

Mining and milling costs in gem mining are so variable, owing to the individual characteristics of each deposit and to varying scales of operation, that such figures are of little value. Premier's costs per load (usually 16 cu. ft. of blue ground in place) from 1927 to 1930 were about 66¢, while those of De Beers for 1926-1927 and 1927-1928 were about

\$1.17. The costs of Cape Coast Exploration, one of the larger of the alluvial mining companies, in 1931-1932 were 66¢ a load, while those of certain other alluvial producers are double this figure.

### CUTTING

Most gem stones before they are used in jewelry are cut or artificially shaped. The art of the lapidary is ancient. It was practiced in Egypt and in the Mesopotamian Valley at least 5500 years ago. The ancient lapidary shaped the stones into scarabs, lenticular or other forms, and then engraved on them characters, figures or scenes. Such intaglios were used as seals. About 500 B.C. fine banded agates began to be cut with figures in relief, each of the variously colored layers being used for part of the design. These cameos were used for decorative purposes only. The only gems the Roman lapidary faceted was the beryl and the emerald, but even to this day the Chinese lapidary, the leader of all sculptors of hard stones in the round, does not facet stones. The cabochon cut, so suited to the star sapphires and rubies, garnet, turquoise, opal, and moonstone, is one species of sculpture in the round still practiced by the Western lapidary. Work in the round, however, fails to bring out the beauty of transparent gems possessing "fire," and gem cutting requiring the faceting of such gems is most effectively done in Europe and in America. The art of faceting was commonly employed in Europe over 500 years ago and is still being improved. It originated, however, in India perhaps 2000 years ago with the crude shaping of cleavage fragments ("lasques") and the polishing of natural crystal faces ("naifes").

In the process of diamond cutting, after careful study the piece of rough is first given an octahedral shape by either cleaving or sawing. Bruting follows, giving the stone the shape of the brilliant with incipient facets. This is done by rubbing two stones against one another. Polishing, by which the facets are perfected and smoothed, follows, and it is done on a soft iron wheel, fed with olive oil charged with diamond dust, the wheel making about 2500 revolutions per minute. Antwerp and neighboring parts of Belgium have some 20,000 diamond cutters; Amsterdam, 5000; Hanau and other near-by German cities 5000. New York, the Jura Mountains, London, Paris and Rio are much less important cutting centers.

Such colored stones as are not cut cabochon are usually fashioned into trap or step-cut gems. In plan this cut is oblong with a table above, parallel to which are several sloping facets down to the girdle; while below the girdle are a number of similar facets, becoming smaller as the culet is approached. The noble gems except the diamond are usually cut in London, Bombay, Colombo or Paris. The cheaper stones, particularly the agate, are cut in Idar and Oberstein, Germany, in the Jura Mountains, France, and in Russia. Czechoslovakia specializes in cutting garnet and imitation stones, and China in jade.

## THE DIAMOND

The diamond, the most desired of precious stones, is commercially by far the most important gem and the only one, with a rare exception, to which modern mining and milling methods are applied. Chemically it is pure carbon, therefore it will burn if heated in oxygen, but aside from this it is an extremely stable body. It crystallizes in the cubic system, the commonest form being the octahedron, the faces of which may be curved and frequently show triangular pittings. The rhombic dodecahedron is also a common form, as are twins according to the spinel law, while cubes are known. The diamond varies from colorless and transparent through slightly tinted yellows, grays and browns to virtually opaque black stones. Deeply colored stones of attractive colors, especially blue, green, or red, are known as "fancies" and are the most valuable of all gems. The diamond is the hardest substance in the world, having a hardness of No. 10 in Mohs' scale, but its hardness varies slightly in different crystallographic directions, and even stones from different diamond fields differ appreciably in this property. It cleaves perfectly parallel to the octahedron and the myth that it will resist the blow of a hammer has on occasions proved to be the most expensive of misstatements. Its rather high specific gravity (fine gems, about 3.52) accounts for its concentration by nature in gravels and by man in his pans and jigs.

The diamond owes its brilliance and "fire" when skillfully cut to its high refractive index and to its dispersion, greater than that of any other colorless gem. As it is a good heat conductor, it is cold to the touch. It becomes positively electrified when rubbed. Some diamonds fluoresce after exposure to light or to an electric discharge and also to the action of radium. Under the latter treatment it may turn green but the stone reverts to its original color on being heated to 500° C., or upon recutting. The diamond is transparent to X-rays, a ready method of differentiating it from paste imitations.

Hindu legends would lead us to believe that the discovery of diamonds in India, the oldest known diamond field, predates the birth of Christ several millennia, but a study of early Oriental commerce suggests that the first Indian stone attracted the eye of some native crossing a pebble-bottomed stream not earlier than 800 B.C. The oldest bediamonded artifact known to us is a little bronze Greek goddess (British Museum, Bronze No. 192) with eyes of diamond crystals dating about 480 B.C. In this and other collections a number of Roman rings set with rough diamonds are preserved but most of these postdate the beginning of our era. The *Artha Sasta* of Kautilya,\* a Hindu work of the early part of the third century B.C., describes six kinds of diamonds from as many mines. Manlius, the Roman poet and astronomer (16 A.D.) was the first Westerner

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\*S. K. Aiyangor: *Quart. Jnl. Mythic Soc.* (1914) 3, 129, Madras.

to describe the diamond even vaguely, but 50 years later Pliny gave what we may call a scientific description of the gem so valuable as to be "only known to kings."

While the Romans valued it as the most precious of all things, the diamond did not really begin to come into its own until the Middle Ages, for not till then had the art of diamond cutting progressed sufficiently to bring out more than a modicum of the stone's fire. Its popularity also grew with the ever increasing improvement in the lighting of homes at night. The increase in wealth has permitted the average citizen of our time to possess a stone which 500 years ago was the insignia of great wealth. Its popularity has further been dramatized by countless gem lovers, notably by Charles the Bold, Agnes Sorel, Cardinal Mazarin, the Empress Eugenie, and, indeed, by every civilized woman. Agnes Sorel was probably the first woman to wear the gem, it having been almost exclusively the ornament of kings and men of wealth before her day.

The production and sale of the gem have increased markedly. It was only in the fifteenth century that its use became relatively common and in the next 200 years India's output increased notably. However, even in the eighteenth and the first half of the nineteenth century, the world's production was worth but from \$1,500,000 to \$4,000,000. In the next two decades, it almost doubled and then South Africa's marvellous mines raised the figure in the late eighties and nineties to \$20,000,000. By 1910 it was \$48,000,000, and previous to the depression 7,000,000 carats, worth at the mine from \$70,000,000 to \$85,000,000, was considered an average year's output. In 1935, the caratage had been regained but as the stones were of poorer average quality, the value was less. If it were possible to fuse a year's production into a cube it would be about 2 ft. 5 in. on edge, while the gravel or rock would be 774 ft. on a side; in other words, about 34,000,000 units of gravel or rock are mined and milled. Furthermore some of the alluvial mines remove  $3\frac{1}{2}$  units of overburden to one unit of gravel treated. The reward seems small, considering that some 15,000 whites and 110,000 blacks, or other natives, are employed.

The world's aggregate diamond production has been about 279,000,000 carats, or roughly 61.5 tons. This is equivalent to a cube slightly over 8 ft. on a side but from this but about  $13\frac{3}{4}$  cu. ft. of fine cut gems of over one carat could be cut. Of the total, by weight, South Africa has produced about 70 per cent, the Congo-Angola field 12 per cent, Brazil 5.5 per cent and Southwest Africa and India each about 4 per cent. As to value, the percentages of India, Brazil and South Africa would be somewhat increased. Until 1870 all diamonds came from placer mines but from 1878 to 1921 the South African pipe mines dominated the production. Since 1907, five remarkable alluvial fields have been found, and through their increasing output since 1922 the production of placer stones has exceeded that from the pipes. In 1932, owing to market



TABLE 2.—*Diamond Production for Typical Years*

Country	1912			1922			1928			1935		
	Carats	Per Cent	Value, <sup>a</sup> Per Cent	Carats	Per Cent	Value, Per Cent	Carats	Per Cent	Value, Per Cent	Carats	Per Cent	Value, Per Cent
South Africa: mines.....	4,888,575	79.1	76.2	405,634	33.0	19.8	2,258,182	29.5	26.9	274,318	3.6	7.6
Alluvial.....	183,307	2.9	8.4	203,925	14.4	29.7	2,114,075	27.8	53.0	402,404	5.3	28.6
Total.....	5,071,882	82.0	84.6	609,559	47.4	49.5	4,372,857	57.3	79.9	676,722	8.9	36.2
Southwest Africa.....	992,380	16.0	11.8	144,156	10.2	8.4	503,142	6.6	6.7	128,464	1.7	4.2
Tanganyika Territory.....							24,681	0.3	0.5	1,415		
Belgian Congo.....				250,292	17.7	9.9	1,647,700	21.6	4.2	3,812,023	50.0	23.0
Angola.....				98,683	7.0	6.9	237,511	3.1	2.6	481,015	6.3	10.6
Gold Coast.....				6,535	0.5	0.1	660,536	8.7	2.8	2,172,563	28.5	11.5
Sierra Leone.....				159,246	11.3	17.6	132,483	1.7	2.5	250,000	3.3	10.1
British Guiana <sup>b</sup> .....	7,800	0.2	0.1	80,000	5.7	7.0	50,000	0.6	1.0	46,564	0.7	1.3
Brazil <sup>b</sup> .....	100,000	1.6	3.4	3,427	0.2	0.6	1,500			50,000	0.6	3.0
Miscellaneous producers <sup>b</sup> .....	6,000	0.2	0.1							3,500		
Total.....	6,173,162			1,411,898			7,630,410			7,622,866		
Value at mine.....	\$57,943,400			\$22,302,970			\$101,690,733			\$29,497,330		
Value per carat.....	\$9.38			\$15.79			\$13.33			\$4.04		

<sup>a</sup> The average value per carat received by the producer (average of world's run of mine for the past 16 years) has varied largely as to the source of rough for that year and the existing basic price of diamonds for that year from \$4.00 to \$27.50, about \$13.00 being a fair average

<sup>b</sup> Estimated.

conditions, the pipe mines were shut down and only now are being reopened. In 1930, the last normal year of production, the alluvial mines accounted for 68 per cent of the production by weight and 55 per cent by value, and that of the underground mines for respectively 32 per cent and 45 per cent. In normal years about two-thirds of the production by weight and three-fourths by value comes from the British Empire. Over 95 per cent of the production comes from the African continent.

The table of the diamond output for the typical years 1912, 1922, 1928 and 1935 (Table 2) shows the changes that have taken place in diamond production in the past 25 years. In part the figures are estimates.

In size, diamonds vary from microscopic crystals to the fist-big Cullinan diamond, which weighed over  $1\frac{1}{8}$  lb. (3106 carats). The average stone recovered weighs from  $\frac{1}{4}$  to  $\frac{1}{6}$  carat, or is about  $\frac{1}{10}$  in. in diameter. India and South Africa are notable producers of large diamonds while the Congo-Angola field rarely produces a 40-carat stone and the largest stone recorded from the Gold Coast is much smaller. Size varies in alluvial deposits in a diamond field and in each pipe, and in cases in different parts of the complex intrusion making up a single pipe.

Similarly, the various diamond fields produce stones of different quality, although there is little if any difference in the better stones from any field. The percentage of fine stones may, however, in one mine be large and in another small. Accordingly at present the run-of-mine stones from one field may be worth but \$2 a carat and from another \$40. On the average the quality of Indian stones was perhaps better than that of Brazil and that of the latter is at least as good as that of Africa.

Each South African pipe produces characteristic diamonds and one familiar with the stones can with certainty, if shown a typical parcel, name their source. The variations are in purity, size, shape and color.

Only a small part of a normal year's production (Fig. 5) is suitable for cutting into fine gems. Almost one-half is bort, material so off-colored, or so badly flawed that it can only be used industrially. A third

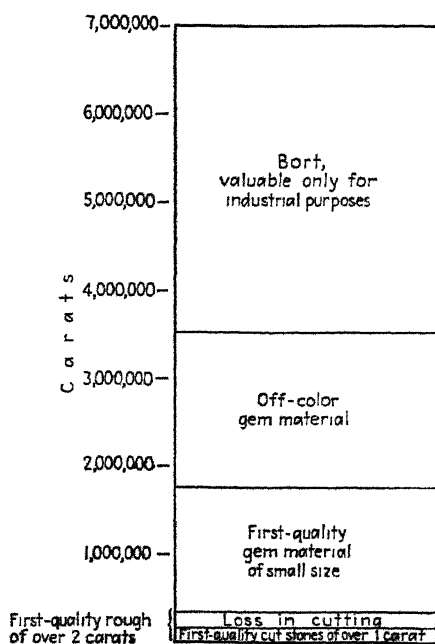


FIG. 5.—MAKE-UP OF WORLD'S DIAMOND PRODUCTION IN A NORMAL YEAR.

quarter is off-color or flawed but can be cut into rather showy jewels eagerly purchased by the Indian, South American and eastern European trade. The fourth quarter is colorless gem material but much of it is small and can be cut only into jewelry mountings. Only about 5 per cent of the total is fine, large rough weighing 2 carats or more, producing after cutting (cutting loss normally about 55 per cent) fine gems of one carat or more. On this 2.5 per cent of the total production, or, say, 150,000 carats, the industry lives. As some of the cut stones exceed 10 carats in weight and many weigh over 2 or 4 carats, there are available for sale each year only about 100,000 fine large diamonds. In consequence, even in 1935, by no means a year of large sales, fine large diamonds were at times difficult to procure.

The known reserves of diamonds are not very large, being of the order of those of gold, zinc or lead rather than that of copper, nickel or coal.

The pipe mines normally have a supply of blue ground partly developed, equivalent to from 3 to 5 years output. The more important mines have undoubtedly a long life ahead of them although with depth it seems to be a rule that the pipes decrease in size, as does on the average the diamond content. Many of the South African alluvial fields have already been exhausted and alluvial mining in South Africa has presumably passed its peak. The Southwest African, the Congo-Angola and the Gold Coast fields have gravel blocked out for a dozen year's operations and will probably not be exhausted for a further 10 to 20 years. Each year as prospecting continues the chance of the discovery of new fields becomes less likely, and such discoveries, rather than the artificial production of the stone, is the menace the industry fears.

#### GEOLOGIC AND GEOGRAPHIC OCCURRENCE

The common occurrences of the diamond is as a pebble in gravel, a secondary source varying from gravel, to thoroughly cemented conglomerates and ranging in age from present river or marine benches to hard pre-Cambrian rocks. The classic example of a source rock from which such pebbles are derived is that of the pipe mines of South Africa, where the diamond occurs as a crystal, in a dark, greenish, basic igneous rock called kimberlite. This occurs as a vertical, steep-sided, funnel-shaped mass of rock, which has been forced from below through a series of flat-lying sedimentary rocks. The cross section of the pipe is as a rule circular or elliptical and its diameter varies from a few tens of feet to about 2300 ft., with the surface exposure from less than an acre to over 80 acres. Kimberlite pipes and dikes occur over a vast area in South Africa, Southwest Africa, Rhodesia, Tanganyika Territory and southeastern Belgian Congo, there being hundreds of them. Some are barren of diamonds, some contain a few but not enough to pay, while the number having a commercial diamond content is very small. At the

outcrop of the pipes that have been worked there was a notable enrichment of diamonds, due to the blowing away of the lighter components of the decomposed rock by the wind. In the unaltered rock in virtually all the pipes the diamond content decreases with depth; that is, the present surface appears to have been the horizon of maximum diamond deposition. Elsewhere diamonds occur in still more basic rocks, in both stony and iron meteorites and they have perhaps been formed artificially in metal melts. In South Africa and in Australia diamond has been found in andesites. Certain of the upland deposits of Minas Geraes, Brazil, are igneous breccias, the highly altered matrix of which presumably is andesitic, if not more acid, in composition. The presence of diamonds in granitic rocks is unproved, but in the writer's opinion is far from impossible. That diamonds may be constituents of some regionally or contact-metamorphic rocks also seems reasonable. In short, the diamond is a mineral of multiple sources although kimberlite is so dominant commercially that its importance is usually exaggerated.

The diamond, while it occurs in only a few places in commercial quantities, has been found in all five of the continents.

*India.*—India produced practically all of the world's diamonds until the second decade of the eighteenth century, when the richer Brazilian mines became the principal producers. In the seventeenth century, India in certain years produced several hundred thousand carats, but its production at present is only a thousand carats or so a year. The Panna district is now the principal producer. All of the Indian stones are secondary, the deposits being modern gravels or very ancient conglomerates of pre-Cambrian age. India, with South Africa, is the producer of big stones and practically all of the historic diamonds are of Indian origin, the Koh-i-Noor, the Regent, the Orloff and a host of others.

*Borneo.*—The fields of Borneo, presumably discovered between 600 and 1000 A.D., were in addition to India the only source of diamonds in the Middle Ages. Late in the seventeenth century, they furnished probably 50,000 carats a year, but their present production is but a few hundred carats a year. There are two regions in which diamonds occur in certain of the river gravels, one in the western part and the other in the southeastern part of the island. The stones are of fine adamantine luster, fair-sized ones being not unusual, and colored ones abundant. The local black diamond is in demand for mourning jewelry throughout the East. Mining is attended by curious rites in order that the spirits of the mine may be favorably disposed to the mine adventurer. Arabs long established in the region cut the stones for the Eastern trade, and since the local supply of rough ones is inadequate, South African stones are imported.

*Brazil.*—Diamonds were recognized about 1720 in the gold placers of Diamantina, Minas Geraes, probably by Bernardo da Fonseca Lobo,

a Brazilian colonial who had lived in India. From Minas Geraes diamond discoveries spread to the near-by states, notably to Bahia, Matto Grosso and Goyaz. The total production for the 210 years of exploitation has been about 16,000,000 carats, which were worth about \$135,000,000. In the heyday of the field, annual production reached 600,000 carats but now is much less.

The announcement of the discovery at Rio in 1729 resulted in a "rush," and the diamondiferous area was rapidly extended. The crown at once prohibited further gold mining and expelled the inhabitants of the district, some 6000 people. Planters, however, were permitted to employ their slaves in diamond mining by paying a head tax. Over-production, which threatened the stability of the diamond market, resulted, and the head tax was greatly increased. The planters made no money but contraband mining thrived and there was no decrease in production. A period of contract mining, 1740-1772, followed, and a single trader was given a monopoly of the field for periods of from three to five years. On Jan. 1, 1772, the government decreed that contract mining should be superseded by state mining and the decline of the Brazilian diamond fields began. State mining proved such a fiasco that in 1834, the year of Brazilian independence, the fields were thrown open to all on the payment of various taxes. Mining is now carried on by prospectors or small groups, but to date no foreign company has been successful, and most have been lamentable failures. The richer African fields proved a bar to larger development of the Brazilian fields.

The diamonds occur in placers, both those of the present stream channels and of ancient elevated terrace gravels, in very ancient conglomerates and also on the uplands in igneous breccias. It should be mentioned that the Agua Suja deposit in Minas Geraes seems to be derived from ultra-basic igneous rocks and that the presence of rocks allied to kimberlite have been reported in the diamondiferous districts. The wide distribution of the diamond-bearing deposits, often in inaccessible districts, leads to much illegal mining, and the workers' low standard of living, together with the presence of many dealers in outlaw stones, has resulted in much stealing at the mines. In certain years, the stolen stones have equaled in quantity and surpassed in quality those declared.

The quality of the Minas Geraes stones is excellent and they are of fair average size, although large ones are rare. When the discovery was made, however, magnates of the diamond industry had considerable stocks of Indian stones on hand, therefore belittled the quality of the Brazilian stones. With the instinct of true traders, the Portuguese sent their diamonds to Goa, whence they were re-exported as Indian stones, and this continued until the prejudice against the Brazilian diamonds disappeared. More than 160 years later, South African stones were shipped to Brazil and re-exported, again to overcome the prejudices of the trade.

Bahia, Brazil, produces, with the gem stones, the world's supply of carbonado (see page 304). This material, once shipped in kegs to Amsterdam as ballast, now sells at the mines for a higher average price than the gem product of any field except that of Namaqualand.

*South Africa.*—The bright eyes of a small boy, Erasmus Jacobs, the intelligent curiosity of a Boer farmer, Van Niekirk, and of a wandering trader, O'Reilly, as well as the mineralogic knowledge of a modest man of science, Dr. W. Guybon Atherstone, led to the discovery of diamonds in South Africa in 1867. Previously South Africa had been a country on the verge of financial ruin, but within a few years immense fortunes were made in the diamond fields and this newly created capital financed the Rand gold field and Cecil Rhodes' diamond-made fortune carried the British flag northward almost to the equator. After the discovery, a few diamond prospectors arrived at the Vaal River, then a sparsely settled semiarid region, but their search was halfhearted. In 1869, however, the "Star of South Africa," a magnificent diamond of  $83\frac{1}{2}$  carats rough and of  $46\frac{1}{2}$  carats cut, was found. While surpassed by many other stones in size, the Star of South Africa is the most important diamond of all time, for it led to the financial regeneration of Africa.

The pipe mines in normal times employ about 18,000 blacks, representing many of the South African tribes. During the rise of pipe mining the alluvial diamond production of South Africa appeared to be stabilized at from 150,000 to 250,000 carats a year, but in 1926 the excessively rich Lichtenburg alluvials were discovered in northwestern Transvaal. A rush of tremendous proportions followed and from 1926 to 1929 the alluvial production was extraordinarily large—from 800,000 to almost 2,320,000 carats a year. The product on the average was poor, resulting in a surplus of small off-color goods. By 1930 this field's production had fallen to normal and in 1935 it was only some 187,000 carats. Alluvial diamond mining in South Africa, conducted as it is by individual diggers or by small, ill financed syndicates, has been on the whole an unprofitable business. The luck of the exceptional miner that strikes a rich pocket or recovers a fine stone is in sharp contrast to the miserable return to the majority. The standard of living of the average digger and his family is desperately low and as, on the average, the more shiftless of the population have drifted into this trade, the solution of the digger problem is one of the most serious with which South Africa is faced.

A few diamonds had been found before the war in Namaqualand, on the west coast of South Africa, south of the mouth of the Orange River. New discoveries were made in 1925 and it was soon realized that in certain of the marine terraces of that desert region there were small local deposits of unequaled richness producing stones of a quality and average size which no other field had ever produced. One of the deposits was in private hands but the title to the mining rights to most of the region

rested with the South African Government, and so state mining began. The state mines from 1925 to 1935 have produced about 1,700,000 carats worth £16,088,000. The 1935 production was 116,496 carats. The Cape Coast Exploration Ltd. operates in the same region.

South Africa had produced (in addition to stolen stones) to Dec. 31, 1935, diamonds to the value at the mine of £313,810,000, or well over one-fourth of its gold production, and almost one-fifth of the total mineral production of the dominion.

*Southwest African Protectorate.*—In 1908 a negro navvy, who had worked in the Kimberley mines, saw in the coastal desert sands of German Southwest Africa, now the Southwest African Protectorate, a diamond glitter. Deposits were found here and there for 275 miles along the coast and a host of German companies sprung up. Speculation on the Berlin Boerse was wild, and production mounted rapidly with certain companies making astounding profits. The Germans tried to establish a self-contained industry but had made peace with the Diamond Syndicate of London before the World War. At present all the operating properties belong to a great English-American company, the Consolidated Diamond Mines of Southwest Africa. The stones of the original discoveries are small but of excellent quality. In the past seven years, larger stones have been found immediately north of the mouth of the Orange River and the company now produces an excellent assortment of gems. The pay gravel on the whole is relatively low grade but locally wind action has caused rich surface concentration, and the pay varies in thickness from a few inches to as much as 30 ft. The source of the diamonds is considered by some to be kimberlite pipes in the interior of the country (such pipes exist but they appear to be barren of diamonds), while others believe pipes are located immediately off the coast. Before the World War, this field reached a peak production of 1,500,000 carats.

*Central African Diamond Field (Congo-Angola).*—In 1907, a prospector of the Belgian-American Forminiere panned a diamond in the middle reaches of the Kasai River, Belgian Congo. In 1913, more important discoveries were made, and at present the five Belgian companies and one, Diamang, in the contiguous part of Portuguese West Africa or Angola, produce over one-half of the world's diamond by weight, although the value is less as much of the company's product is largely fit only for industrial purposes. In part, the production is of excellent quality. The stones average from 5 to 12 per carat. Diamang produces the larger stones.

As in other alluvial fields, the deposits from the surface down consist of overburden, pay gravel and bedrock. The pay may be from 3 to 7 ft. thick and from 1 to 4 cu. m. of overburden is removed for each cubic meter of gravel milled. The gravel averages about one carat per cubic meter. It occurs in stream beds, in river flats, in terraces more or less

elevated above the present streams and in the beds of an ancient river system crossing the present drainage. The original source of the diamonds is unknown but the pay gravel is apparently concentrated from certain gravel lenses at the base of the flat-lying Lubilache sandstone, a Jura-Trias formation. The diamonds, like those of Gwelo Forest, Rhodesia, originated long before the intrusion of the late Cretaceous kimberlite pipes of South Africa. To this earlier generation of diamonds, those which are from time to time recovered at the Rand gold mines may belong, but on the other hand the latter stones, of undoubted pre-Cambrian age, may be very much older and representatives of a third period of diamond formation.

*Gold Coast.*—In 1919, Sir A. E. Kitson, head of the Colonial Geological Survey, found diamonds on the Birrim River, Gold Coast. At present, the Gold Coast produces from 1,500,000 to 2,000,000 carats yearly, the principal producer being the Consolidated African Selection Trust. The stones are small (15 to 25 to the carat), but of good quality, and many of them are used in drilling.

*Sierra Leone.*—In 1930, officers of the Geological Survey found two small diamond crystals in the colony of Sierra Leone. The stones are of good size and consist of both fine gem material and bort. The Consolidated African Selection Trust has a monopoly of the field, which it works through its subsidiary, Sierra Leone Selection Trust. Exports began in 1932 and by 1935 the production is believed to have reached some 250,000 carats. The gravels are reported to be rich and the field appears to be of first importance.

*British Guiana.*—Diamonds were found in British Guiana in 1888 by Edward Gilkes, an intelligent colored gold prospector. The stones are small but of excellent quality and the annual production has varied from about 10,000 carats to 214,000 carats, with a present rate of 40,000 carats. The deposits appear to be of relatively small tonnage and are more adapted to exploitation by the "pork-knockers" than by companies.

*Miscellaneous Sources.*—Minor diamond fields have at one time or another been exploited in Tanganyika Territory, Rhodesia; Shan-tung, China, Venezuela, French Equatorial Africa and Australia.

Diamonds have been found in a number of localities in the United States. They occur as mineralogic curiosities in Virginia, North Carolina, Georgia, and other southeastern states; a number have been recovered from gold sluices in California and isolated finds are reported from Oregon and several of the Rocky Mountain states. Near Murfreesboro, Ark., is a stock of kimberlite, which has produced several thousand diamonds, but as yet there is no proof that the deposit is commercial. In the terminal moraine of the last glacial epoch in Wisconsin, Illinois, Indiana, Michigan, and Ohio, diamonds have been picked up here and there by the merest chance. The stones are of excellent water and of



fair average size. There is, therefore, to the north in Canada an original source of fine diamonds, although of course we are ignorant as to whether it carries enough stones to pay.

#### RUBY AND SAPPHIRE

The ruby and sapphire are respectively the red and blue transparent varieties of corundum, crystallized alumina. "Sapphire" also includes all other colors except red, that color being placed before sapphire. Formerly misleading names, "oriental topaz," "oriental emerald" and "oriental amethyst" were applied to these gems. As the dispersion of the corundum gems is low, they lack "fire," but they are valued for their fine colors. "Pigeon's blood" red, and a deep cornflower blue are preferred. Ruby of fine quality rarely occurs of any appreciable size but fine sapphires of considerable size are relatively common. Both ruby and sapphire are characterized by parallel fibrous structures, usually only observed by the expert, and from it he can usually tell the gem field from which the stone is derived. When this structure is highly developed and the stone is cut cabochon, a white six-pointed star floats in the stone. Such stones are called star sapphires, rubies, or asteria. The color is normally bluish gray or grayish pink and fine reds or blues free of a milky caste are rare.

The finest rubies come from Burma. The mines, which were worked at least as early as the fourteenth century, center about Mogok. Ruby and spinel occur in a contact-metamorphosed limestone associated with gneisses and schists, the complex being intruded by both acid and basic granular igneous rocks. The limestone is too poor to work but talus gravel derived from it and the flooring of caves in it may be commercial. The principal source is "byon," a pebbly clay usually covered with some 15 ft. of overburden. The layer next to bedrock is frequently rich. An English company, Burma Ruby Mines Ltd., worked the field from 1889 to 1931, but while its production was relatively large (for example 1923-1924 value of stones, £53,747), the enterprise was on the whole unprofitable. The present production by natives is relatively small. Besides rubies and sapphires, spinels, blue tourmalines and a number of other gem stones are recovered as by-products at Mogok.

Ceylon for at least 2500 years has produced sapphires and some rubies and from there, and to a lesser extent from Burma, come star sapphires and rubies. The rubies are generally pale in color but the sapphires are frequently fine, although paler than those of some other gem fields. The gems occur in the gravels of broad stream beds, the original source of the ruby and some of the sapphires being crystalline limestone while others are derived from gneisses and schists. The gravel in instances extends to a depth of 120 ft. Gems are especially abundant in the lower gravels, particularly in depressions in the bedrock. Spinel, chrysoberyl,

topaz, zircon, garnet, tourmaline and a number of other gem stones are by-products of sapphire mining.

Southern Siam and contiguous parts of Cambodia have gem fields that produce sapphire and some rubies, the latter having a purplish brown tint. Although the stones, as they are exported via Siamese ports, are sold in Europe as Siamese stones, the majority come from Cambodia. This region, once an important producer, is reported to be approaching exhaustion. Rubies occur in crystalline limestone in Afghanistan and Badakshan.

Fine, deep blue sapphires occur in a pegmatite dike in the Zanskar range, Kashmir. First discovered in 1908, it was energetically worked from 1924 on, the production being in certain years several million carats. Sapphire occurs in the gravels of New South Wales at several places, notably in the Inverell district. The Anakie field in Queensland is more important, however. The sapphires are inky blue and appear in artificial light almost black: yellow or sage green sapphires also occur, and an occasional ruby. The stones occur in old gravel deposits elevated above the present stream beds. The field was discovered in 1870 and reached its peak production (\$320,600) in 1920.

Rather pale sapphires with a steely luster, and also rubies, are recovered from stream gravels in Granite and Powell Counties, and near Helena, in Montana. They were also mined from a monchiquite-camptonite dike in Yogo Gulch, Fergus County, Montana. Operations ceased in 1929. Several hundred thousand carats were produced annually some 10 years ago but only one-fourth was of gem grade, the rest being used industrially, largely for watch jewels.

#### EMERALD, AQUAMARINE, YELLOW BERYL AND MORGANITE

These gems are all members of the beryl family, a silicate of beryllium and aluminum, which characteristically crystallizes as six-sided prisms. It is fairly hard (7.5 to 8), but its other physical characteristics are not striking, and its gem varieties owe their beauty to their color and transparency.

The deep grass green, velvety variety of emerald is the most valuable of all precious stones, but probably no unflawed emeralds of fair size exist. Practically all fine emeralds come from the Muzo or Cosquez mines in Colombia, both owned by the government, which from time to time has farmed them out. The Muzo mine, by far the more important, occupies a precipitous valley slope in an isolated region 90 miles north-northwest of Bogota. Emerald with calcite and a number of other gangue minerals occurs in veins of pneumatolytic origin, which cut in all directions highly folded black carbonaceous shales, presumably of Lower Cretaceous age. The Chivor deposit is somewhat similar, although albite and quartz are the principal gangues and the color of the gems is not so

fine. At many of the other emerald occurrences, the gem occurs in schists near pegmatitic intrusions, the causative rock. Such are the Ural Mountain, the Egyptian, the Habachthal (Austria), the South African and the North Carolinian occurrences. Some of the Australian deposits are of this type but the gem also occurs in pegmatite dikes. The emeralds at Bahia, Brazil, occur with quartz and calcite in cavities in crystalline limestone. Next to those at Muzo, the Russian stones are best, but they do not compare with the Colombian in fine deep color.

The other gem varieties of beryl, aquamarine, the sea green variety, golden beryl, the yellow, and morganite, the rose pink, occur as constituents of pegmatites or in gravels derived therefrom. Brazil, Madagascar, Southwest Africa, the Urals, the Transbaikial district (Russia) and India are the principal sources.

#### CHRYSOBERYL AND ITS VARIETIES, CAT'S-EYE AND ALEXANDRITE

Chrysoberyl is a pleasing greenish yellow gem. Cat's-eye is a variety containing many parallel fibers, which, when the stone is cut cabochon, exhibits marked chatoyance. The groundmass of the gem is honey (greenish) yellow and the "pupil" whitish. Alexandrite is a most intriguing gem, being bluish green or dark green by natural and raspberry red by artificial light. The Ceylon gravels are the most productive field of all three varieties, but alexandrite was first found in the Urals and Brazil produces chrysoberyl. The stones are of pegmatitic origin, although the immediate host rock may be gneiss or schist.

#### OTHER GEM STONES

*Spinel.*—Spinel has two gem forms, the rose red balas ruby and the spinel ruby of ruby color. Indeed, the Black Prince's ruby, in the English Regalia, which has not without reason been considered by some as the most beautiful gem in the world, is in reality a spinel ruby. Ceylon, Burma, Afghanistan and Siam are the principal localities. Spinel usually occurs in contact-metamorphic limestones or in gravels derived from them.

*Spodumene.*—Spodumene has three attractive gem forms: (1) the rare yellowish green hiddenite from North Carolina; (2) the more common yellow from Brazil, and (3) the rose or lilac-pink kunzite from near San Diego, Calif., and from Madagascar. Because of the perfect cleavage the gem is difficult to cut. It is of pegmatitic origin, although some of the gems are recovered from gravels.

*Opal.*—The opal is a transparent to translucent gem traversed by myriads of tiny fissures, or tiny veinlets of later opal, which reflect and refract the light, resulting in a lovely play of all the colors of the rainbow. The ground may be colorless to almost black, the latter type being particularly esteemed. Hungary (now Czechoslovakia) was once the

principal source of opals but the Australian gems are much finer than the Hungarian. The yearly output of Australia (largely New South Wales and South Australia) 15 years ago was worth \$225,000 but is less now.

*Topaz*.—Topaz shows some "fire" and the colorless blue, sherry yellow and pink varieties are rather attractive gems. The rose pink variety is usually a yellow stone changed in color by careful heating. Brazil and Siberia are the principal sources. The gem occurs usually in pegmatites or in gravels derived from them.

*Peridot*.—Peridot is the transparent olive green variety of olivine and chrysolite, the greenish yellow variety. While an attractive gem, it is too soft to be used where subjected to much wear. The finest come from Zebirget Island in the Red Sea.

*Tourmaline*.—Tourmaline appears in practically all colors and is frequently particolored at right angles to the main axis of the prism or zonally parallel to that axis. The deep green and rose pinks are particularly attractive. Maine, California (near San Diego), Brazil, Madagascar, Burma and Russia are the principal producers. The gem is typically a pegmatitic mineral, although it is also recovered from gravels.

*Zircon*.—Zircon, because of its high refraction and great dispersive power, has next to the diamond the greatest "fire." It occurs colorless ("matura diamonds") and yellowish red (jacinth). Certain brown Siamese stones when heated change to a sea green-blue. Siamese, Ceylonese and Australian gem gravels are the principal sources of supply.

*Garnet*.—The garnet family includes several members which are used as gems, notably pyrope (blood red with a yellowish tinge); almandine (violet-tinged crimson); hessonite (orange yellow); demantoid (emerald green, with highly developed "fire"); and spessartine (orange red). They occur in a variety of igneous and metamorphic rocks and in gravels and in many different countries. The attractive raspberry red rhodolite from North Carolina should be mentioned.

*Turquoise*.—Turquoise, although opaque, is attractive because of its robin's-egg blue color. Probably the first large-scale mining was that of turquoise in the Sinai Peninsula. The stone had also been a great favorite of the American Indians for centuries before the white man arrived on the continent. Persia and our own Southwest are the principal sources of supply.

*Jade*.—Under the name "jade," two distinct but quite similar minerals, nephrite and jadeite, are included. An emerald green is the most desired color, although it occurs also in other shades of green as well as yellowish and black. The stone varies from translucent to opaque. Always a favorite in China and India, it became popular in the West a generation ago. The principal localities of nephrite are Chinese Turkestan, Russia and New Zealand, and that of jadeite, Burma. Nephrite occurs in regions of metamorphic rocks and jadeite as an albite-jadeite intrusive rock: both occur as stream gravels.

*Lapis Lazuli*.—Like turquoise, lapis lazuli is opaque and its beauty depends upon its color, a deep Prussian blue. Afghanistan is the principal locality.

*Amber*.—Amber is the fossil resin of a pine tree and was one of the earliest decorative substances used by man. Its pleasing yellow transparency is well known to all. It is extensively mined on the Prussian coast and occurs in many other parts of the world.

*Quartz and Feldspar*.—Space permits only mention of the more beautiful members of the quartz family: the limpid rock crystal; the purple amethyst; the yellow citrine; the apple green, translucent chrysoprase; the red carnelian and the banded agate. Among the feldspars the moonstone with its intriguing opalescence, labradorite with its varying iridescent blues, grays and greens, and the green amazonstone are the finer varieties.

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## CHAPTER XVII

### GRAPHITE

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**G**RAPHITE is a crystalline form of carbon, chemically the same as diamond and charcoal. Because it was mistaken for lead, it was called plumbago and black lead, and graphite pencils are still almost universally called "lead pencils." In 1789 Werner proposed the name "graphite" from "grapho" meaning "to write."

Graphite is separated into two classes, natural and manufactured (originally termed "artificial"). The two are unlike in many physical properties and yet for certain purposes can be substituted for each other. The coarser natural crystalline varieties have never been duplicated in the laboratory. Further, natural graphite is divided into two classes, which, unfortunately, have been designated as "crystalline" and "amorphous." The material classified as amorphous is extremely fine and has a dull gray, lusterless appearance, but the individual grains possess a crystalline structure just as definitely as the variety called crystalline, so that is a misnomer. The only distinction between amorphous and crystalline is one of relative grain size. Unscientific as this classification is, it has been useful in the trade and has become so firmly fixed that doubtless it will continue. There is seldom any difficulty in making the separation, as intermediate specimens are uncommon.

#### OCCURRENCE AND ORIGIN

*Geologic Occurrence.*—The general statement can be made that graphite is found in nature in igneous, sedimentary and metamorphic rocks. In the igneous rocks it has been found in iron-nickel meteorites, in effusive and plutonic rocks, in dikes and in pegmatites. In the sedimentary rocks it is sometimes noted as small detrital particles, which have been transported and deposited along with other clastic minerals, but because of its softness it is apt to be ground to such small size that it is generally unrecognizable in the sedimentary rocks. The most common occurrence is in the metamorphic rocks, especially the gneisses, schists and metamorphosed limestones. In these the graphite rarely constitutes more than 6 per cent of the rock, although it may make up practically the entire rock. It also occurs in quartzites.

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Graphite exists in the rocks in numerous different forms. In the schists and gneisses it is present in small, thin flakes seldom over  $\frac{1}{8}$  in. in diameter. These are oriented parallel to the foliation and are disseminated through the rocks, as are the mica flakes in the mica schists. In the limestones the graphite flakes are similar in character but seldom show marked parallelism of orientation. In the igneous rocks the graphite is also in the form of unoriented thin flakes. In the pegmatites the flakes are normally much larger, even up to an inch or more in diameter, and also are considerably thicker. In the igneous and pegmatitic occurrences, the graphite is occasionally present in pockets or lumps, some with radiating structure. Graphite also occurs in the form of veins in which other minerals may be entirely lacking. These massive occurrences are rare except in Ceylon. They commonly show foliation but do not readily split into thin flakes or fibers. Another important type of occurrence is in the form of beds that are readily recognized as former coal strata now altered to graphite.

*Origin.*—Much has been written concerning the origin of graphite but there still remain many unanswered questions. A number of purely hypothetical explanations have been offered.

When beds of coal have been intruded by dikes or sills of igneous rock and the coal converted into amorphous graphite for some distance from the contact, there can be little doubt as to the origin. The volatile materials of the coal have been driven off by the heat of the lava and the remaining amorphous carbon converted into the crystalline condition. Some of the best examples of this type occur near Raton, N. M.,<sup>9</sup> and in Sonora, Mexico.<sup>8</sup>

Some of the most important graphite deposits of Canada are believed to have been formed by contact metamorphism where granite and syenite have intruded Grenville limestone. The graphite has been produced by the decomposition of carbon dioxide, which was either an igneous emanation or was derived from the limestones.\*

Flake graphite in the gneisses, schists and marbles has generally been explained as formed by dynamic metamorphic action from organic matter previously present in the rocks and derived from either plants or animals. Whether the hydrocarbons were broken up, causing the precipitation of the carbon direct, or CO and CO<sub>2</sub> were first formed and they in turn decomposed by the withdrawal of the oxygen and the precipitation of the carbon, is not known. In either case the graphite was not formed *in situ* but moved to concentration centers where the crystalline

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<sup>9</sup> References are at the end of the chapter.

\* M. E. Wilson: *Geology and Mineral Deposits of a Part of Amherst Township. Mem. 113, No. 96 Geol. Ser., Can. Geol. Survey (1919). 54 pp.*

Mineral Deposits in the Ottawa Valley. Can. Geol. Survey, *Summary Rept.* (1919) pt. E, 19-29.

flakes were formed. It has been suggested that the carbon may have been derived from carbonates,  $\text{CaCO}_3$ ,  $\text{MgCO}_3$ ,  $\text{FeCO}_3$ , etc., which were broken up by intense heat and pressure to form CO and  $\text{CO}_2$ , which in turn were decomposed.\*

When graphite has been found in igneous rocks the carbon is believed to have existed as original gases of hydrocarbons or as oxides of carbon, all of which are present in volcanic lavas, or to have been derived from carbonaceous rocks cut by the igneous intrusions. When graphite is found in pegmatites or other dike rocks it is believed that the carbon was picked up from underlying carbonate or carbonaceous sediments. The formation of crystalline flakes of graphite from coke in the blast furnace is familiar to all metallurgists, and may explain the natural process of graphite formation in the igneous rocks. Veins of graphite are also thought to have been formed in some places by the transformation of some of the solid bitumens.

#### DISTRIBUTION OF GRAPHITE

##### *United States*

Graphite is widespread in its occurrence. It has been reported from nearly every state within the United States in which there are any metamorphic rocks. At one time or another it has been mined in Alabama, Alaska, Arkansas, California, Colorado, Connecticut, Georgia, Idaho, Maine, Massachusetts, Michigan, Montana, Nevada, New Hampshire, New Jersey, New Mexico, New York, North Carolina, Pennsylvania, Rhode Island, South Carolina, South Dakota, Texas, Virginia, Washington, Wisconsin, and Wyoming. Its presence has been noted in Oregon and Vermont, although, so far as known, no attempts have been made to mine it in these states. A product called graphite was also mined in Ohio in 1904. Both the amorphous and crystalline varieties have been produced in the United States and of the crystallines both the flake and the massive vein types. The flake variety is most abundant and wherever worked is contained in gneisses and schists.

The states where flake graphite has been most extensively mined are Alabama, New York, Pennsylvania and Texas. The conditions of occurrence in all these states are so similar that it is unnecessary to give detailed descriptions of each.

Alabama graphite operations were begun in 1888 but were of little consequence until 1913. During the World War there was marked activity in the region, with 30 plants in operation during 1918 and 10 others near the operating stage. The demand for domestic graphite to replace the material from Ceylon and Madagascar, and the high prices

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\* A. N. Winchell: Petrographic Studies of Limestone Alteration at Bingham. *Trans. A.I.M.E.* (1924) 70, 884-903.



prevailing, were responsible for this development. Since 1918 the industry has declined in spite of strenuous efforts to revive it. At present all the Alabama plants are closed and most of the mills in ruins.

The graphite belt of Alabama, with the exception of a 10-mile break between Goodwater and Millerville, extends continuously northeast-southwest for 60 miles, with a maximum width of 5 miles. It is contained in Clay, Coosa and Chilton Counties. The graphite-bearing rock is a mica schist containing numerous pegmatites. The original rock is thought to have been sandy shales with some interbedded sandstones. The commercial graphite rock occurs in bands varying in width up to 100 ft. but generally 20 to 60 ft. The minerals associated with graphite are quartz, sillimanite, feldspar, muscovite, biotite, pyrite (limonite in altered rock), apatite and garnet. The graphite flakes average about 1 mm. in diameter and  $\frac{1}{15}$  mm. in thickness but range from microscopic size to 5 mm. or even more. The pegmatites also carry graphite; in places even more than the country rock. The average graphite content of the beds worked seemed to be between 2.5 and 3.5 per cent. Some companies have claimed an average of 4 to 5 per cent but in some cases rock as low as 2 per cent C was used. The beds are unusually regular and persist for miles with a fairly regular southeast dip of  $30^{\circ}$  to  $45^{\circ}$ .

The Alabama graphite beds carry much pyrite in the unweathered rock, which has a bluish tint. They are deeply weathered, the pyrite being altered to limonite and the feldspar to kaolin, so that the rotten rock is of a rusty color. The average depth to the hard rock is 30 to 50 ft. The graphite flakes in the fresh rock are tougher than in the weathered portion. Practically all work has been confined to the weathered material.

As to origin of the Alabama deposits, Prouty<sup>14</sup> says:

Since the graphite is most commonly found in the beds which were originally more siliceous, the conclusion that it was originally in the form of petroleum or petroleum products seems more justifiable than that it was in a finely divided form of carbon such as one meets in the carbonaceous shales.

The New York graphite deposits are located in the Adirondack region and contained within Essex, Warren, Saratoga, Washington and St. Lawrence Counties. Alling<sup>1</sup> enumerates 26 localities of mines and prospects in the first three counties. They are most numerous in the vicinity of Ticonderoga, where the first mine was opened soon after 1850. Few of the numerous companies have been successful even for short periods and there has been practically no production since the close of the World War.

In the northern portion of the state the graphite is commonly found at the contact of sedimentary and intrusive igneous rocks, especially crystalline limestones of the Grenville formation and pegmatites. In

the southern section the graphite is mainly obtained from bedded quartz schists or feldspar-quartz schists that carry from 5 to 7 per cent of graphite.

Graphite deposits are present in several places in southeastern Pennsylvania, but principally in Chester County, where the industry was prosperous for several years. Practically nothing has been done since the close of the World War. The graphite is obtained from graphitic schists associated with crystalline limestones of pre-Cambrian age. Pegmatites are numerous and contain much graphite. The deposits are closely similar to those of Alabama.

The Texas graphite deposits are confined to the pre-Cambrian rocks of the Llano-Burnet region, where ancient sediments have been altered to schists and marbles. They have been cut by numerous pegmatites. In some places the schists contain from 10 to 14 per cent graphite. For a time since the World War this section led in production of flake graphite in the United States but all operations have now ceased.

California contains some flake graphite deposits, mainly in Los Angeles County<sup>3</sup> but production has always been on a small scale. In 1935 this region produced the only flake graphite in the entire country.

Vein graphites are poorly developed in several of the flake-graphite regions but a mine near Dillon, Mont., is the only one in the country that has ever produced graphite of the Ceylon type. The veins vary in thickness up to 16 in. The production has always been small.

The amorphous varieties of graphite have been mined in several places to supply small quantities for local consumption for foundry facing and for paints. At Cranston, near Providence, R. I., part of a coal bed has been metamorphosed to an impure graphite, which has been sold to foundries. A graphitic slate near L'Anse, Baraga County, Mich., has furnished a low-grade product for paints. Another amorphous graphite deposit that has been worked on a small scale for many years is a few miles south of Carson City, Nevada.

In most of the other graphite regions of the United States graphite operations have been small and sporadic even though the supply is large and the quality fair.

#### FOREIGN COUNTRIES

Almost every country in the world has at one time or another produced graphite, but a comparatively small number of countries has dominated the world industry, and among them the two islands of Ceylon and Madagascar are most important.

*Ceylon.*—The graphite deposits of Ceylon were reported as early as 1681 but not until 1827, when Joseph Dixon imported some Ceylon graphite to mix with clay for crucibles, do we hear of any production. Since that time there seems to have been almost continuous production and until the World War Ceylon graphite completely dominated in the

crucible field and was extensively used in other lines. In recent years this supremacy has been challenged by Madagascar. The Ceylon graphite deposits are confined to a rather large area in the southwestern part of the island. The product is unique in that it occurs in definite fissure veins cutting ancient gneisses of both igneous and sedimentary origin. The steeply dipping veins vary in width up to 6 ft. and continue downward without any marked change to greatest depth reached, which is about 600 ft. The veins run from northeast to southwest, approximately parallel to the foliation of the rocks. The graphite occurs mainly in the form of large, thick plates irregularly arranged but in places in a fibrous form producing what is called needle lump. In the body of the vein the graphite is remarkably pure although quartz and pyrite may be present in subordinate amounts. Feldspar, apatite, rutile, biotite, pyroxene and other igneous minerals are present in small amounts. In Ceylon flake graphite also occurs in the gneisses and associated marbles but the operations have been exclusively confined to the veins.

The Ceylon graphites have generally been interpreted as of igneous origin, perhaps formed from gaseous emanations, but many other hypotheses have been offered. The mines are mainly small and shallow and the mining operations crude. Hundreds of mines have been opened and abandoned when they reached a depth where the mine waters became excessive. No one knows the reserves of the island but they are probably very extensive and could furnish a greatly increased production if needed and if the mines should be equipped with pumps and other mining machinery of modern type.

*Madagascar.*—In 1908 Madagascar began the production of flake graphite in a small way. It did not receive much attention for several years, then the amount increased to such an extent that it began to compete actively with the graphite of other countries, and in 1917 for the first time it exceeded the production of Ceylon. This one country is almost entirely responsible for the virtual death of the flake-graphite industry of the United States and has affected the industry in all countries of the world.

The graphite deposits of Madagascar are extensive, in an area somewhat east of the central part of the island. The country rock consists of schists and gneisses in part of which the flakes of graphite constitute a large percentage. Shelly says:<sup>16</sup>

The graphite occurs disseminated in flakes or lenticles or compact in veins, pockets, masses, or beds, one form succeeding the other without any regular order, but generally without the continuity being broken. The stratification, if such it may be called, is most irregular, being twisted, contorted, folded, and faulted in all directions, adding considerably to the mining difficulties. The deposits lie at all angles of inclination, but in many instances seams and masses of considerable extent, and several feet in thickness, are found lying almost horizontal. The number of deposits is quite remarkable, and they contain practically an inexhaustible supply of ore. They extend from the north of Tananarive to the south of Fianarantsoa, a distance of about 400 miles. So far only the outcrops of superficial

deposits have been opened up, but it is reasonable to infer that deposits exist also in depth. Work has been confined to these surface deposits for the usual economic reasons. . . . The deposits occur in rock which is decomposed to a great depth by atmospheric and chemical influences, which have converted them to the consistence of clay, rendering the extraction and subsequent treatment easy.

*Chosen (Korea).*—Graphite occurs in numerous places in Chosen and has been extensively worked since the Japanese have been in control. Although the bulk is consumed by Japan, some of the product enters the world markets. The major portion is of the amorphous variety extensively used in foundry facings, paints and stove polish but each year there is a small production of crystalline flake graphite. Little information is available concerning the distribution and character of the deposits, but they are reported as extensive and easily worked.

*Canada.*—Graphite deposits are widespread in Canada. They have been worked mainly in Ontario and Quebec but prospected or worked on a small scale in Nova Scotia, New Brunswick, British Columbia, Labrador, and on Baffin Island. The New Brunswick deposit consists of graphitic slates containing amorphous graphite. In Ontario and Quebec, where most of the mining has been done, the crystalline flake variety is developed in graphitic schists and limestones. Many mines have been worked for varying periods but in general they have met with the same obstacles as those in the United States.

The most remarkable deposit in the country is near Calabogie, Renfrew County, Ont., where the Black Donald mine has long been operated. At present it is the only active graphite mine in Canada. The deposit has the form of a vertical body varying from 15 to 70 ft. in width and has been worked to a depth of over 200 ft. Calcite is the principal impurity present although chlorite and some other silicate minerals are present in small amounts. The walls consist of Grenville crystalline limestone and sericite schist. The graphite is thought to be a metasomatic replacement of a part of the limestone by contact metamorphism. The flakes are all small. Where the mined rock contains 60 per cent of graphite, about 15 per cent of the concentrates will be classed as flake graphite and 45 per cent, composed of extremely fine flakes, is termed dust or amorphous. In recent years some of the Black Donald graphite has gone into crucibles and pencils but most of it has been sold for the cheaper products.

*Mexico.*—For a number of years Mexico has furnished a considerable amount of amorphous graphite from the southern part of the State of Sonora. The graphite has been formed by the metamorphism of Upper Triassic coal beds, which have been intruded by dikes of igneous rocks. The beds range in thickness up to 24 ft. For some time material averaging 86 per cent C was worked, with some specimens as high as 95 per cent, but in recent years the grade has been lower. It is shipped to the United States and thence distributed to the trade. For many years it found

large application in the pencil trade but recently it has been consumed mainly in foundry facings and for dry batteries.

*Germany.*—The Passau graphite deposits of Bavaria have long been extensively worked. During the Middle Ages, some of the alchemists' crucibles are said to have been made of this material. During the World War, the production in 1917 amounted to 42,825 tons. Although it has greatly declined, the product is still extensively mined. The graphite is of the crystalline flake variety and is disseminated through schists and gneisses. It is mainly recovered from the decomposed rocks. The product is used in almost all kinds of graphite manufactures.

*Czechoslovakia.*—The graphite deposits of Bohemia and Moravia are a continuation of the Passau deposits. The production has been large for many years, amounting to 41,113 tons in 1927.

*Austria.*—The Austrian graphite deposits are similar to those of Bavaria and Czechoslovakia. Before its dismemberment, Austria had the greatest production of graphite that any country has ever had—59,530 metric tons in 1917. Austria and Germany supplied the needs of the Central Powers during the World War when other sources of supply had been cut off.

*Russia.*—Russia has never been an important producer of graphite but recently it has been preparing for an extensive production. Under the U. S. S. R., 26 widely separated localities in both European and Asiatic Russia have been thoroughly investigated and three mills have been built, one of which is said to be the largest graphite plant ever constructed. The country contains various grades of graphite, both amorphous and crystalline.

*Other Countries.*—At various times many other countries have produced graphite. Among them the most important are Italy, Spain, Norway, Sweden, France, Switzerland, India, Japan, South Africa, South Australia, New Zealand and England.

#### POLITICAL AND COMMERCIAL CONTROL

Graphite is one of the minerals of international trade. Before the World War practically all the graphite crucibles in use for melting steel and brass were made from Ceylon lump graphite and German clay, and it was generally believed that the Ceylon material was essential for the manufacture of high-quality crucibles. The World War made changes necessary but on its conclusion again Ceylon graphite was demanded. However, experimentation has continued until at present throughout America and Europe crucibles of better quality than before the war are being manufactured with a minimum amount or even a complete absence of Ceylon graphite. For several years manufacturers, both in America and Europe, regarded Mexican graphite as the best material for the highest grade of pencils. Here, too, other materials are now in use

without affecting the quality of the pencils. With new and improved methods for refining and grinding graphite, substitutions became feasible and the cost is the determining feature in the choice.

With the wide distribution of graphite throughout the world, the political and commercial control in case of war cannot present serious difficulties to many countries. Germany and Austria proved their independence of Ceylon graphite during the World War, and the United States was well started along the same line. Nevertheless, Great Britain and France, in possession of Ceylon and Madagascar, still possess a commercial advantage.

In the United States, possessing almost inexhaustible supplies of graphite and extensively engaged in the manufacture of graphite commodities, we have seen mine after mine close in recent years, until production has practically dropped to nothing and is now almost entirely confined to the operation of a few low-grade amorphous deposits in which the product is used for foundry facings and paints. Tariffs have been levied and increased by Congress and yet the domestic product has not been able to compete with the product from Madagascar, Ceylon, Chosen and Mexico. The explanation lies in the cheaper production of the higher grades of graphite in these foreign countries. Recently the tariff on Madagascar graphite has been materially lowered in a reciprocal trade agreement with France. Germany, Austria, Russia and Japan are now endeavoring to make themselves independent of all other graphite-producing regions.

#### PRODUCTION AND CONSUMPTION

Fig. 1 shows the varying annual productions of the leading graphite-producing countries of the world. The increase between 1914 and 1919 is explained by the employment of graphite in the manufacture of war supplies. The general increase between 1925 and 1929 shows increased industrial activity, as does also the general upward trend during the past few years. Some of the drops in production of individual countries are to be explained by tariffs and the substitution of one graphite for another owing to changing costs or changing needs of graphite manufacturers.

The principal consumption of graphite has always been confined to the industrial nations of the world. Among these are the United States, England, Germany and France, with Japan and Russia rapidly entering the same class. The introduction of graphite crucibles for the steel industry just over 100 years ago marked the beginning of active graphite production and consumption. Previously the amount used in pencils, paints and small crucibles for the laboratory was extremely small. As the machine age has developed, the requirements have become ever greater and the upward trend may be expected to continue during normal times.

## PROSPECTING, EXPLORATION AND MINING

Graphite is so easily recognizable that even the ordinary layman could scarcely fail to identify it. It may be confused with molybdenite, but a simple chemical test for sulphur or the streak on a piece of porcelain should be sufficient for an exact determination. The streak of molybdenite is bluish to greenish and that of graphite a dull black.

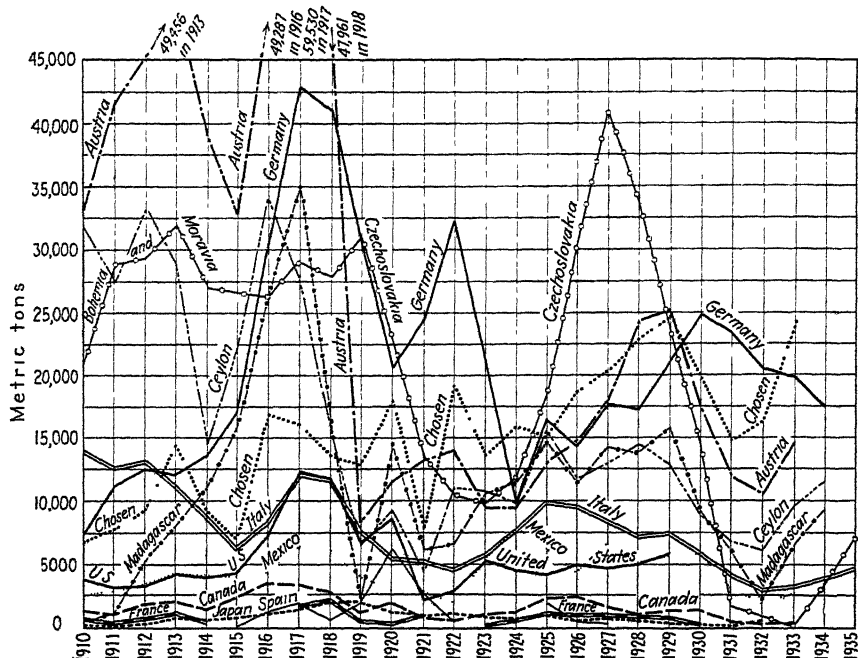


FIG. 1.—PRODUCTION OF GRAPHITE IN PRINCIPAL COUNTRIES, 1910-1935.

When a deposit of graphite is found, it is important to determine the percentage, the extent and regularity of the deposit and the quality of the graphite. The associated minerals are also important, as the presence of other flaky minerals, such as mica, may result in difficulties of separation.

No unusual mining methods are employed in the graphite mines, nearly all of which are comparatively shallow.

## PREPARATION FOR MARKET, TESTS AND SPECIFICATIONS

For most purposes the graphite rock as mined contains too large an admixture of other minerals to permit its use in the raw state. Occasionally the graphite content is unusually high and the use does not demand a high-grade product. Both the character of the graphite and the intended use determine the method of beneficiation to be employed. For foundry facings and paints, considerable amorphous graphite is used in which the only preparation consists of fine grinding. Sometimes such material is screened, to remove some of the alumina and silica minerals. Ceylon

graphite is refined by breaking the lumps by hand and removing the quartz and other minerals. The fine-sized associated minerals are separated by winnowing.

The disseminated flake graphite, which is most widely distributed and most generally used, calls for special methods of preparation. Usually this type of graphite constitutes only a small percentage of the rock, and the associate minerals, mainly quartz, calcite and aluminum silicates, must be separated and discarded.

The first step in concentration of flake graphite is to crush the rock to such a stage as to break the bond between the graphite and the other minerals. With rotten rock, this is simple, because weathering agents have done most of the work in the alteration of the feldspar to kaolin, the pyrite to limonite, and the removal of considerable material by solution. Very little force is necessary to free the graphite flakes.

Various types of ore-crushing machinery have been used in the reduction of the hard fresh rock. There seems to be some preference for rolls, as it is claimed that they do not break the flakes as badly as other crushers. For the finer grinding, ball mills or rod mills are generally used.

It is common to treat graphite in two stages, concentration and refining. The concentration processes raise the carbon content to 80 to 85 per cent whereas the refining may bring it up to 90 to 95 per cent, or in exceptional cases even higher. In the process of concentration, crushing is followed by separation of the graphite flakes in a variety of ways. Log washers were the first machines used and proved to be fairly satisfactory, as the thin graphite flakes are readily floated in agitated water and removed in the surface runoff. Many other "skin-flotation" devices using the same principle have been employed, such as buddles, concentration tables, various kinds of float boxes and oscillators. Some kerosene added to the water has helped to keep the flakes at the surface, to be floated off. Pneumatic processes and dry tabling for separating the flat flakes from the more angular particles of the associated minerals have been tried but were not successful. Electrostatic methods have been used in some mills, by which the feed was passed through an electric field and the graphite particles with higher electrical conductivity were deflected and fell into a compartment separate from the impurities.

The most efficient methods for concentration of flake graphite, and those mainly installed in the more modern plants, embody froth flotation. Several different flotation methods are known to have been used. Probably no two mills and no two flowsheets are alike, but each new installation is based on experimentation with the particular type of rock. The flotation of graphite involves no principles or processes not used in other types of mineral flotation.

The concentrate obtained in the first beneficiation lends itself to further refinement by the elimination of fine quartz and various silicate minerals that may be present between the laminae of the graphite flakes



or attached to the outside of the flakes. This is accomplished by fine grinding of the concentrates, particularly by means of rolls or buhrstones, and followed by screening. Naturally, the graphite flakes are apt to be reduced in size, which is undesirable, but this disadvantage is more than compensated by the improved purity of the flake.

At best, the physical methods for graphite-flake beneficiation cannot furnish a pure product. In some instances chemical methods have been used by which the remaining amounts of silica and silicates are dissolved by the use of hydrofluoric and other acids. It is possible in this way to obtain practically pure graphite but necessarily at an almost prohibitive cost.

*Tests and Specifications.*—When graphite is offered for sale, it is necessary to know first the percentage of carbon and second the amount and character of the impurities. Ordinarily the carbon content is determined by combustion, although there are a number of other analytical methods. The determination of the mineral impurities can be made by microscopic examination with a petrographic microscope or by ordinary chemical analysis. The specifications for graphite depend almost entirely upon the chemical and physical properties and vary with the use for the material. In general, refined graphite contains 90 per cent or more of carbon.

#### MARKETING, USES AND PRICES

The marketing of graphite usually involves several operations. Some large consumers buy from the producers and in their own plants refine or blend to secure the grade demanded in their manufactures. The more usual method is for certain firms to engage entirely in buying, importing, refining and blending, and supplying the material to the manufacturers under the specifications agreed upon. This is generally much more satisfactory, especially for small consumers.

The uses of graphite are numerous and of extremely varied character. The use in pencils probably antedates all others. The modern pencil is made of the best and softest graphite, in which gritty minerals are almost entirely absent. The graphite is finely ground, mixed with a fine quality of clay, molded into shape and baked. The hardness of the pencil depends upon the amount of clay present and the temperature at which it has been baked. Mexican graphite long dominated the pencil trade but it is now being replaced by a higher carbon graphite which, by modern methods, can be reduced to the necessary fineness.

The crucible trade at one time consumed the larger part of the commercial graphite but this is no longer true because crucible steel is being largely replaced by the electric-furnace product. Some crucibles are still being used in the steel industry but most of them are employed in the melting of brass. For nearly 100 years Ceylon graphite was considered necessary for crucibles but recently it has been supplanted by flake

graphite from various countries. The best material for mixing with the graphite is the clay from Klingenberg, Bavaria. During the World War domestic clay was used in the United States.

The softness, greasy feel and flaky character of graphite render it desirable for lubricating purposes. Although occasionally used dry, it is generally mixed with oils. Graphite for this purpose must be free from grit. Practically all kinds of high-grade natural as well as manufactured graphite can be used. Graphite lubricants are especially recommended for heavy machinery.

Graphite paints are largely used for metal work. The lower grades are satisfactory, provided that impurities, such as pyrite, which undergo oxidation changes, are absent.

A very large part of the commercial graphite is used as foundry facing. For this the cheaper and less pure varieties are employed, particularly the amorphous type. In the electrical industry graphite is utilized for dynamo brushes, for electrodes, for dry batteries and other objects, because of its high conductivity. In this field the manufactured graphite competes with the natural product.

Amorphous graphite is employed in the manufacture of stove polish. Graphite is used for glazing powder. It has also a great variety of other minor uses.

*Prices.*—The market prices of graphite have always been difficult to determine because there are so many varieties and grades. Most sales are individual transactions based on quality. The demands for particular uses and the supply naturally affect the prices. In general three classes of graphite are quoted: Ceylon lump, chip (smaller sized particles), and dust; Madagascar (also Canadian or domestic) flake; and amorphous graphite, particularly that of Chosen and Mexico. The two crystalline varieties are quoted by the pound and the amorphous by the ton.

Before the World War, Ceylon lump sold for 7¢ to 10¢ per pound, chip 4¢ to 6¢ and dust  $2\frac{1}{2}$ ¢ to 4¢. During the war the prices were increased about 300 per cent, so that at one time in 1917 the best grade of Ceylon lump sold for 32¢. Since the war the prices have receded to about prewar prices or slightly less. Madagascar and other flake graphites of 85 per cent C or higher normally sell for 5¢ to 7¢ per pound, but these prices also were extremely high during the war, some selling at 18¢. Amorphous graphite, according to grade, normally sells for \$10 to \$30 a ton, but unusually good material has gone as high as \$50 a ton.

#### MANUFACTURED GRAPHITE

In 1896 Edward G. Acheson obtained a patent on a process for making graphite in the electric furnace. It has been called "artificial graphite" but is more properly named "manufactured graphite." It is produced by heating a mixture of some carbonaceous material such as anthracite

culm or petroleum coke with smaller amounts of quartz sand and sawdust. It is claimed that carbides of silica, iron, etc. are first produced. These are decomposed at temperatures of about 7500° F. and the impurities volatilized, leaving a mass of practically pure amorphous graphite. The first plant was at Niagara Falls but now a number of plants both in the United States and abroad are engaged in the manufacture of the product.

Manufactured graphite finds wide application in industry and for a number of uses is in sharp competition with natural graphite. It is used chiefly in lubricants, dry batteries and electrodes but there are numerous minor applications.

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## CHAPTER XVIII

### GRANULES

By H. HERBERT HUGHES,\* MEMBER A.I.M.E.

**G**RANULES, or roofing granules, as perhaps they are more commonly known, include crushed slate, "greenstone," quartzite, slag, brick, and other rocks or ceramic products used either in their natural state or artificially colored. Crushed slate has been widely used by roofing manufacturers, and all granules are commonly called "slate" irrespective of their composition.

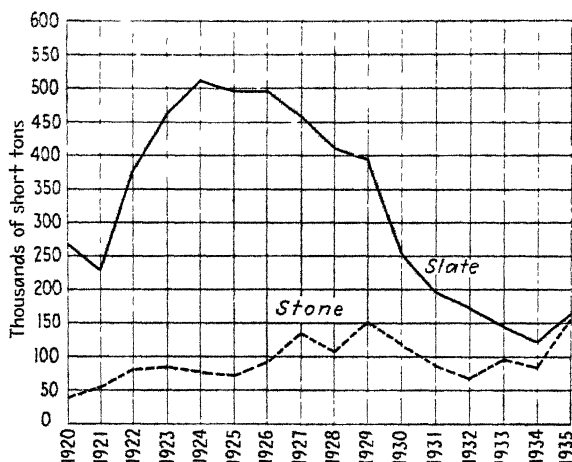


FIG. 1.—SLATE AND STONE GRANULES SOLD OR USED IN THE UNITED STATES.

Data are from the U. S. Bureau of Mines and do not include crushed brick, slag, glass, burned clay, or glazed crushed gravel. Slate flour is included with granules from 1919 to 1928, inclusive.

Roll roofing antedates asphalt shingles, although both are made of a layer of roofing felt impregnated with an asphaltic compound. The original purpose of surfacing materials on roll roofing was to prevent sticking in the roll, and large quantities of talc and mica are still used for this purpose on both roll roofing and shingles. The fire-resisting properties of a thicker surface coating were recognized during the early years of roofing manufacture, as was also the protection afforded the asphalt from the actinic rays of the sun. Granules, or rather the forerunner of present-day granules, primarily served a utilitarian purpose. The development

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of granules for color and decorative effects parallels the growth of asphalt shingles.

Fundamentally, the manufacture of roofing granules is closely allied with production of crushed stone and might well be included with that industry if it were not for the wide variety of materials employed. Slate, greenstone, and other rock granules, whether marketed in a natural state or treated, are merely carefully graded products of crushed-stone operations. "Silicate" or "ceramic" granules for the most part are also crushed-stone products subjected to further processing. On the other hand, no close relationship exists between slag or glass granules and the crushed-stone industry.

In a typical granules plant primary reduction is by jaw or gyratory crushers, after which the material passes through rolls and thence to screens. The average product is the fraction that passes 8 mesh, or possibly 10 mesh, and is retained on 35 mesh. In some slate-granules plants, fines are pulverized and sold as slate flour, and various sands for industrial or abrasive uses may be produced at plants grinding quartzite or quartz gravel for granules. For the most part, however, fines have little value and accumulate in huge waste piles. Care is taken to keep the finished granules free from dust. Elaborate dust-collecting systems in many plants serve the twofold purpose of protecting health and ensuring a clean product. In some instances natural rock granules are oiled before shipment. This procedure not only eliminates dust but also temporarily brightens the color.

#### HISTORICAL DEVELOPMENT

Berger<sup>1</sup> has published an interesting account of the development of the granules industry, from which much of the following information is taken. Asphalt shingles were introduced about 1907, through the simple procedure of cutting roll roofing into shingle-like strips. The common surfacing materials in use at that time were fine gravel and feldspar chips which, although durable, resulted in monotonous, light gray roofs that soiled easily.

About the same time the slate industry was becoming concerned over the high percentage of waste in slate quarrying, amounting to at least 80 per cent of the slate quarried. In collecting statistics of the slate industry for 1909, the U. S. Geological Survey asked quarrymen whether any disposition was made of the waste. About 95 per cent of the producers replied in the negative, although nearly all indicated that an outlet for waste would be of great value to the industry. This interest in waste disposal logically led to production of slate granules. In 1912 it was reported that some of the waste slate was broken into small pieces and sprinkled on an asphaltic surface for use on roofs, although the actual

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<sup>1</sup> E. H. Berger: *Ceramic Roofing Granules*. *Ceramic Age* (1935) 25, No. 2, 49.

introduction of slate granules was probably as early as 1909 or 1910, and the first mill expressly for the production of slate granules was erected at Granville, N. Y., about that time.

Separate statistics on slate granules were first published in 1919, when 202,611 short tons valued at \$1,155,140 were reported, representing nearly 20 per cent of the total value of all slate produced during the year. The peak of slate granules production was reached in 1924 with an output of 512,810 short tons valued at \$3,178,454, amounting to 27 per cent of the total value of slate produced. Statistics for these earlier years include slate flour, but in 1924 it amounted to only about 5 per cent of the material included as granules. In 1935 slate granules, exclusive of flour, produced in the United States amounted to 165,000 short tons, valued at \$1,126,000, or 31 per cent of the total value.

Green always has been the most popular color for granules. Green slate, however, is rather dull except when wet, and other green rocks came into use about the same time as slate. Various rocks containing epidote, amphibole, or other green minerals have been used for granules, and each has commonly been termed "greenstone." Paints and oils have been employed to improve color, but such superficial treatment has little permanent value.

Architects and builders demanded brighter and more permanent colors for granules. Crushed waste brick, tile, pottery, and other ceramic products were used in a small way about 1912 for buff and red shingles, but because of high porosity and poor adhesion to the asphalt they were not entirely satisfactory. The first artificially colored granules on which the color was permanent were patented in 1916. They were made by granulating blast-furnace slag in a sodium silicate solution. Chrome or iron oxide was then used to coat the silicate-slag granule and the material was heated until the silicate coating fused sufficiently to absorb the color.

Glass granules made by fusing limestone and sand colored by oxides were introduced in 1921. They were nonfading and nonabsorbent and gave excellent results in concrete mosaics. For roofing, however, their semitransparency, conchoidal fracture, relatively poor adhesion, friability, and rather high cost were undesirable.

Granules coated with "silicate" were developed about 1925 or 1926. Slate granules were wet with a sodium silicate solution containing pigments of various kinds and colors. After drying, the granules were heated sufficiently to sinter the silicate and make it translucent but not enough to form a transparent glaze. Sodium silicate films formed by this process on the proper base apparently will last for years, for granules of this type have been in use for about 10 years and have been weather-resistant. An early objection to silicate granules was their tendency to get a white coating or "bloom" in damp weather when the temperature was about 50° F. This coating would be removed by the first heavy rain-

fall, but in the meantime the appearance of a continuous heavy frost was not desirable. Fortunately, in recent years processes have been developed to prevent "blooming."

Experimental work in producing bright-colored "ceramic granules," particularly those coated with a vitreous enamel, was begun about the same time that silicate granules were developed, and at present granules made by a similar process are widely used. Crushed quartz, low in iron, is treated with a coloring agent and a weatherproof complex silicate vehicle and fired in a rotary kiln at a temperature that may run as high as 2400° F. but not high enough to fuse the granule proper. The insoluble glaze is impervious and highly resistant to weathering.

Since 1929 granules have been made by burning sized No. 3 Pennsylvania clay or Watsonstown shale. Shades of buff and red or even other colors may be produced through the addition of metallic salts that oxidize upon firing. This burning after sizing is a relatively new idea in granules made from clay or shale. The red product is used extensively in asphalt-brick siding with the buff to represent mortar joints. The shape of the granules is good; they adhere to the asphalt properly and weather well.

#### PRICES AND PROPERTIES

The rapid growth of the slate granules industry was due in part to low costs resulting from utilization of waste material at roofing-slate operations. The average value per ton f.o.b. plant in 1919 was \$5.70. It was somewhat higher during the next few years, but in 1924, the year of peak production, it was only \$6.20. In 1935, the value f.o.b. plant ranged from about \$7 a ton to nearly \$8, although some apparently was sold at lower prices. The strong demand for brighter colors, however, has resulted in a thriving business in ceramic-type granules at prices averaging about \$12.50 a ton f.o.b. plant. More than half the granules now used by roofing manufacturers are the more expensive bright colors, with shades of green predominating.

Irrespective of cost, granules should be opaque, or nearly so, rather than transparent, in order to retain their proper color when embedded in the asphalt. They should be relatively nonabsorbent to resist freezing and thawing without disintegration or spalling. Efflorescence or blooming is undesirable, but this disadvantage of some of the earlier artificially colored granules apparently has been overcome. Good adhesion to the asphalt coating is essential under all weather conditions. Granules should not be brittle but should be tough enough to withstand abrasion during handling and shipping.

Proper grading of the granules is important, and care must be taken to avoid segregation of sizes during shipping and in storage. The exact size and grading of commercial granules vary within the limits of minus 8 mesh to plus 35 mesh. Specifications commonly call for not more than

1 to 15 per cent retained on 10 mesh and not more than 5 to 10 per cent passing through 28 mesh.

## MARKETS

Virtually the entire output of granules is consumed in the manufacture of prepared roofing, including roll roofing, shingles and siding. The prepared-roofing industry held up remarkably well during the depression period following the financial panic in 1929, the low in 1931 amounting to 57 per cent of the 1929 peak whereas the low of building contract awards in 1932 was only 17 per cent of the 1929 peak. Prepared roofing, of course, is widely used in the maintenance of all types of structures, and owners kept roofs watertight even though other repairs might be delayed.

Complete production data on all types of granules are not available, but the U. S. Bureau of Mines figures on slate and stone granules since 1919 are summarized in Table 1. The sharp rise in output of stone

TABLE 1.—*Slate and Stone Granules Sold or Used in the United States, 1919-1935\**

Year	Slate			Stone		
	Short Tons	Value		Short Tons	Value	
		Total	Average per Ton		Total	Average per Ton
1919	202,611	\$1,155,140	\$5.70	26,167	\$112,332	\$4.29
1920	268,516	2,044,942	7.62	40,000	240,000	6.00
1921	231,770	1,897,886	6.03	54,393	343,735	6.32
1922	379,980	2,177,061	5.73	82,410	593,432	7.20
1923	462,260	3,268,554	7.07	86,000	630,000	7.33
1924	512,810	3,178,454	6.20	77,370	527,656	6.82
1925	497,700	3,210,904	6.45	72,560	596,217	8.22
1926	498,050	3,009,368	6.04	91,530	626,583	6.85
1927	459,760	2,775,962	6.04	136,340	858,630	6.30
1928	413,980	2,468,471	5.96	107,830	667,435	6.19
1929	395,830	2,321,330	5.86	153,870	931,781	6.06
1930	255,070	1,549,302	6.07	117,510	727,264	6.19
1931	198,450	1,182,684	5.96	87,960	546,320	6.21
1932	174,140	1,058,713	6.08	68,960	458,795	6.65
1933	146,880	1,024,917	6.98	97,680	720,356	7.37
1934	123,290	902,078	7.32	85,410	687,336	8.05
1935	165,000	1,126,000	6.82	159,110	775,241	4.87

\* Source: U. S. Bureau of Mines. Figures do not include brick, slag, burned clay, or glazed crushed gravel. Slate flour is included with granules from 1919 to 1928, inclusive, but in 1923 and 1924 available data show that it amounted to only about 5 per cent of the total. Data from 1919 to 1923 were collected by the U. S. Geological Survey.

granules reflects the demand for brighter colors, and the tonnage for 1935 is made up largely of artificially colored granules. A large part



of the slate-granule output is also treated to brighten the natural colors. The total of 324,110 short tons of slate and stone granules in 1935 does not include burned clay, slag, glass, or glazed crushed-gravel products.

Canadian plants produced about one-third of the apparent consumption of 18,115 short tons of granules in Canada in 1935; the rest came from the United States. Natural green rocks predominated, although various shades of slate, quartz and crushed brick were also produced.

The granules industry has made rapid strides since 1930 in color-processing its products and quarrying and milling practice have kept pace with the crushed-stone industry.

## CHAPTER XIX

### GYPSUM

BY D. H. NEWLAND\* AND H. J. BROWN,† MEMBERS A.I.M.E.

#### NATURE, ORIGIN AND DISTRIBUTION

BY D. H. NEWLAND

**G**YPSUM, which finds wide use in the cement and plaster industries, is one of two calcium sulphate compounds found in nature. Its formula is  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ . When pure it contains 32.5 per cent calcium oxide, 46.6 per cent sulphur trioxide and 20.9 per cent water. The crystallized mineral, known as selenite, if free of inclusions, may show on analysis approximately the theoretical percentages. Most rock and earthy sorts of gypsum, such as supply our main industrial requirements, carry admixed clay, silica, iron oxides, lime and magnesia carbonates and other impurities, so that their composition departs considerably from the theoretical. Material with 90 per cent or more of hydrous calcium sulphate is satisfactory for the generality of uses; much of the commercial rock is below rather than above that limit. Very exceptional in quality is rock that carries no more than one per cent or so of foreign matter. Gypsum of such grade, when fine-grained and compact, is called alabaster. It finds special applications in industry as terra alba and for calcined plasters and has ornamental uses.

Anhydrite, the second calcium sulphate mineral, lacks water, as the name indicates, and consists of calcium oxide 41.2 per cent and sulphur trioxide 58.8 per cent. It is an inert substance, resistant to chemical change, and is regarded as an impurity when present in gypsum. It is frequently associated with gypsum as an intergrowth or as included discrete bodies. It also occurs independently in extensive deposits.

#### PROPERTIES

Gypsum is monoclinic in crystal habit. The crystallized mineral found in cavities, veins and as disseminations in clays, shows generally a simple combination of forms, but may be prismatic, flattened or acicular in shape, depending on the relative development of the faces. The fibrous, pearly variety is known as satin spar. Cleavage is perfect in the plane of the clinopinacoid and the cleaved sheets resemble colorless mica, but, unlike the latter, are only slightly flexible and altogether

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inelastic. Gypsum is softer (hardness, 2) and lighter in weight (sp. gr., 2.3) than calcite or limestone. Rock gypsum, when free of moisture, weighs from 140 to 150 lb. per cu. ft. A bedded deposit averages about 3000 tons to the acre for each foot of thickness.

Anhydrite, in contrast to gypsum, is orthorhombic in crystal system, cleaves in three directions at right angles to each other and is considerably harder (hardness, 3 to 3.5) and heavier (sp. gr., 2.9), outweighing even limestone to the cubic foot. Notwithstanding these distinctive properties, by which it may be readily recognized, there is no doubt that anhydrite has often been mistaken in the field for gypsum, especially in the records of well borings. For that reason, principally, it has been regarded as a rather uncommon mineral and often little note has been made of its occurrence; yet anhydrite deposits are even more widespread than gypsum, especially below the subsurface.

Gypsum is slightly soluble in water, somewhat more so than anhydrite. At 0° C. one part dissolves in 415 parts water, whereas at the same temperature one part anhydrous sulphate requires 525 parts water. The solubility of both minerals increases with temperature and at 38° C. the ratio for gypsum is 1:368 and for anhydrite 1:466. From about that point the solubility decreases until at 100° C. the ratio is 1:452 for gypsum and 1:572 for anhydrite. There are metastable phases that complicate the solubility relations. In the presence of alkaline chlorides the solubility is measurably increased. Hydrochloric acid is the usual solvent in the laboratory.

Tests indicate that the compressive strength of rock gypsum ranges from about 2600 to 3200 lb. to the square inch.

Exposed to weathering and erosive agencies at the surface gypsum succumbs rather rapidly, more rapidly than limestone, so that the presence of gypsum beds under earth cover is often indicated by an irregular hummocky condition. Solution cavities and widened joints or mud seams characterize rock gypsum in the ledge, to the extent at times of seriously complicating quarry operations or involving excessive waste from decomposed and discolored rock. On the other hand gypsum is easily drilled and broken and the expense of extraction is usually low.

#### ORIGIN AND MODE OF OCCURRENCE

Commercial gypsum deposits for the most part belong to the sedimentary class in which the mineral occurs in beds or layers intercalated between limestones, shales and sandstones, or more rarely with rock salt. In such association they usually have been considered, doubtless rightly so, as precipitations from the evaporation of saline waters in basins in which the shales, limestones or rock salt were laid down. The saline waters may have existed as a salt lake, or they may have been a part of the sea shut off from the open ocean by a bar or enclosed as a gulf.

Sea water of the present day holds calcium sulphate to the extent of 3.6 per cent of the total solids. In process of evaporation, calcium carbonate precipitates first, then calcium sulphate, next rock salt and last, on final evaporation of the solution, the potassium and magnesium compounds. The normal arrangement for bedded marine deposits thus calls for limestone at the base and salt in the roof of the calcium sulphate, but this order is not necessarily always characteristic of field occurrences. Re-solution of the more soluble compounds and shifting about of the deposits before consolidation and final burial may lead to a varied order, even within the limits of a single basin. Several beds of gypsum and anhydrite characterize the succession in certain large basins like the Salina of the Eastern and the Permian of the Western States.

It appears from experimentation that either gypsum or anhydrite is precipitated from solution according to the prevailing temperature and the presence or absence of other dissolved compounds. In saline solution the precipitate will be gypsum if the evaporation is carried on below about 30° C.; above that temperature, anhydrite. What the temperature may have been in past geologic ages when the deposits were formed is unknown, but there is little doubt that anhydrite is prevalent in many districts and is the more persistent form of calcium sulphate under conditions of rock pressure and temperature that obtain at moderate depth. Gypsum is more often found on the surface, because it is the more stable mineral under atmospheric conditions. It can be shown that some of our most important bedded deposits have a zonal arrangement, with gypsum on the outcrop and for some distance below ground and anhydrite at greater depth. The change from one form to the other is likely to be gradual, a zone of mixed gypsum and anhydrite intervening between the two.

Some tests have been made to determine the feasibility of converting anhydrite into gypsum on a commercial basis. In finely ground condition, anhydrite will slowly dissolve and crystallize as gypsum, but the process is hardly practicable on a working scale. The experimental work of M. Farnsworth in the Bureau of Mines showed that anhydrite in particles ranging from 7 to 18 microns in diameter required from 3 to 18 weeks to complete the change.

It is characteristic of water-laid calcium sulphate to be persistent in the plane of bedding and to be fairly uniform as to chemical and physical features over considerable stretches. At some places a seam 4 or 5 ft. thick may be traceable for miles and show little change in composition throughout its extent. Such consistency of character is important when the gypsum is to be used in the manufacture of calcined plasters, for the setting qualities of these depend in great measure upon composition.

Secondary calcium sulphate may originate through the interaction of sulphuric acid or other sulphate compounds with calcium carbonate, either in disseminated form or as solid beds of limestone. Thus are formed

scattered crystals of gypsum in clays and occasional more substantial bodies that may simulate bedded deposits, but which seldom are of commercial grade. Such bodies are likely to be irregular in shape and to exhibit marked changes in composition from place to place. An undisturbed sedimentary environment in general affords little opportunity for the development of extensive bodies of secondary gypsum, yet these may attain some importance in folded and faulted strata, especially in the vicinity of mineral lodes that carry pyrite or other metallic sulphides.

Surficial accumulations of unconsolidated gypsum, admixed with clay and marl, are found in many of the western and southwestern states. This material is extensively employed in wall plaster. This soft gypsum, or "gypsite," as it is called, is found in lowlands and old creek channels, usually in the vicinity of beds of rock gypsum or anhydrite. The deposits result from the solvent and transporting activity of circulating ground waters that come in contact with calcium sulphate in their movement. The gypsum is formed as an efflorescence or by subsurface evaporation of the waters. The deposits are commonest in dry regions where the rainfall is intermittent. Shallow and local as a rule, occasionally they reach a thickness of 20 or 25 ft. and spread over an area of hundreds of acres. The stucco made from the calcined product of gypsite lacks the whiteness of that made from high-grade rock, but often it shows exceptional plasticity.

Wind erosion in desert regions results in the formation of gypsum sands by wearing away exposed ledges of rock or breaking up surface concentrations. The Tularosa desert, New Mexico, with its shifting dunes of brilliant white gypsum, is a familiar example. It is estimated that the dunes cover 270 square miles, affording an inexhaustible supply of pure gypsum except for slight contamination with salt.

#### DISTRIBUTION OF DEPOSITS

Fairly widespread as a mineral, and originating under varied conditions, gypsum in bodies of workable grade and quantity is mostly confined to certain horizons or positions in sedimentary environments; in other words, it is a primary accumulation, to be regarded as a unit in the stratified succession. Relatively few workable bodies have originated by secondary processes. Geologically, the sedimentary deposits range from early Paleozoic to Quaternary, but more commonly they characterize formations not older than the Carboniferous. Some are found in proximity to salt beds; others are independent of such association. In most regions the beds have been but little disturbed by regional forces and are scarcely affected by metamorphism in the usual meaning of the term.

#### UNITED STATES

Taken as a whole, the gypsum deposits are unequally distributed over the United States; in the region east of the Mississippi River they

are few and far apart, whereas to the west they occur in hundreds of localities and some single districts spread over many square miles. Notably important are the deposits in the Great Plains region in Iowa, Kansas, Oklahoma and west central Texas. The Rocky Mountain region, from Montana on the north to New Mexico and west Texas on the south, has inexhaustible resources, as yet only partly explored. Gypsum occurs abundantly in Utah and Nevada, less so perhaps in Arizona and southern California. East of the Mississippi only four states—New York, Ohio, Michigan and Virginia—have commercially workable deposits, but the eastern districts rate high industrially because of their market advantages.

The easternmost gypsum deposits of the country are those of central and western New York, where beds of both gypsum and anhydrite accompany the Salina formation of the Silurian system. The Salina otherwise is composed of limestone, shale and extensive rock-salt layers, the latter found only below the outcrop under heavy cover. The formation outcrops as a belt on a line somewhat south of Lake Ontario and the Mohawk River. Fully hydrated rock exists at the surface and for the first 100 or 150 ft. depth; below the material is in the form of anhydrite. The workable deposits range from 4 or 5 ft. to 30 ft. thick, although the better grade of rock is found only in the thinner seams of the western districts in Erie, Genesee and Monroe Counties. Of late production has been restricted to these counties, where underground mining is the method of extraction. Wall plasters, block, tile and wallboard are manufactured. A large tonnage of crushed rock is sold to Portland cement plants in New York and Pennsylvania for use as a retarder.

The Silurian strata extend west from New York into Ontario, where they contain workable gypsum beds, as well as into Ohio and Michigan. In Pennsylvania, which holds the continuation of the Salina strata on the dip, they are too deeply buried to be tapped by shafts and no doubt the calcium sulphate mineral where found is anhydrite rather than gypsum.

In Michigan the calcium sulphate deposits of Silurian age generally are concealed below Devonian and Carboniferous rocks, which spread over all the central counties, but they appear at the surface in the vicinity of St. Ignace, northern Michigan, and on some of the islands in Lake Huron. Around Detroit rock salt is mined from deep deposits of Silurian age which are accompanied by heavy anhydrite strata. The gypsum that is extensively worked in Michigan is found in the Grand River Valley around Grand Rapids and in the Saginaw Bay region near Alabaster. The beds belong to the lower Carboniferous or Mississippian system. Grand Rapids and vicinity is a leading center of the gypsum industry, with several beds from 5 to 20 ft. thick, or more, which yield a good grade of rock for manufacturing purposes. The Grand River Valley, along which the mines are situated, has been eroded down to about the gypsum horizon, so that the beds are easily accessible. The supplies are ample

for the immediate future, although it is not known how far the hydrated rock extends under the higher ground with thicker cover. In the Saginaw Bay districts of Iosco and Arenac Counties, the gypsum also occurs in several beds, but only the uppermost directly below the soil has been extensively worked, by quarry methods. It is estimated that from 40 to 50 square miles of these counties is underlain by gypsum. Some parts yield rock of alabaster quality.

The principal deposits in Ohio are found along the north and south shores of Sandusky Bay, near the western end of Lake Erie. Port Clinton is the chief mining and manufacturing center. One or two beds, from 4 to 8 ft. each, are present in the Monroe formation of Silurian age, akin in stratigraphic position to the Salina beds farther east. They are reached at shallow depths on the bay shores, which accounts for the location of the mines. Elsewhere in Ohio gypsum is reported in many of the central and northern counties at varying depths from the surface. The operations at Port Clinton have been on an extensive scale, the products including cement rock, calcined plasters and other articles, for the markets of Ohio, Illinois and Pennsylvania.

Among the Appalachian States south of New York, gypsum in workable amount is restricted to a single district, in Smyth and Washington Counties, southwestern Virginia. The productive horizon is the Maccrady formation of the lower Carboniferous, composed mostly of shales and limestones that have been folded into a syncline and cut off on the southeastern margin by a great fault. The gypsum is largely in blocks and lenses that are massed along the fault plane; it is believed to be of secondary origin, in part at least, deposited by waters circulating along the fracture, which have derived their calcium sulphate content from disseminated occurrences in the shales. Anhydrite is present in depth and the same beds carry salt. The output is sold to cement mills; it is calcined to plasters and has an outlet as agricultural (land) plaster, for which there is demand among peanut growers of the state.

Between the Mississippi River and the Rocky Mountains the Permian red beds are widely represented, with gypsiferous strata present over extensive stretches. An interrupted series of such deposits can be traced from west Texas across Oklahoma and into Kansas, constituting perhaps the largest resources in contiguous areas in the country. In central Iowa the series seems to reappear after a considerable hiatus and to be represented by substantial deposits around Fort Dodge, the northernmost of the Permian occurrences and a leading district for gypsum mining and manufacturing. The geographic position and excellent shipping facilities contribute to the importance of this area. The resources are provided by a bed, 10 to 30 ft. thick, exposed along the Des Moines River and its tributaries over an area estimated at 40 square miles for recoverable rock. Extraction is by underground mining under advantageous conditions and at low cost. Calcined plasters, wallboard, tile and block are made on a

large scale. The occurrence of gypsum in lower Carboniferous strata at Centerville, southern Iowa, may be noted as a matter of general interest. The deposit is under heavy cover and carries anhydrite.

In Kansas the Permian forms a belt crossing the state from northeast to southwest a little to the east of the middle part. The northernmost commercial district is that of Blue Rapids, Marshall County, where 8 or 9 ft. of gypsum of excellent grade is present. Calcined products are the chief item in the industry. Farther south are many gypsum outcrops, particularly in the counties of Salina, Dickinson and Marion to the south of the Kansas River. Mines have been worked at Hope and near Solomon, but more important as a producing center is Medicine Lodge, Barber County, on the Oklahoma border. This district covers a large part of Barber County and extends well into Comanche County on the west and Kiowa County on the northwest. Several beds of rock gypsum are associated with shales, clays and sandstones, and in part is very pure, suitable for Keene's cement and white finishing plasters.

Wide areas in western Oklahoma are underlain by Permian gypsum strata, the exposed edges of which are displayed as white bands in the bluffs that extend for miles along the Cimarron, Canadian, Washita and other rivers. Mine developments are confined to a few places that have shipping advantages, as the local markets do not suffice to sustain a large industry. Most activity has been shown in Woods County, in the northwest; Blaine County, in the west central part; and Grady County, in the southwest. With extensive outcrops and the periodic nature of the rainfall, the conditions favor the formation of surface deposits of gypsite, which is found particularly in the southwestern districts in Greer and Jackson Counties but are much more important in Texas.

In the State of Texas the Permian beds outcrop in force in the Red River Valley near Quanah, Hardman County, and extend from there in a broad belt west of south to the headwaters of the Colorado River in west central Texas. They include as many as four or five layers of rock gypsum of workable size, besides having local accumulations of gypsite on the surface. Rock is mined mostly for Keene's cement and special grades of calcined plasters, whereas gypsite is preferred for ordinary wall plasters because of its plastic qualities. Another large area of Permian strata with gypsum occurs in Culberson County, east of the Guadalupe Mountains, southwest Texas, next to New Mexico, into which the belt extends. Also, in the Malone Mountains, Hudspeth County, southwest of El Paso, gypsum is exposed in great abundance and has been used in cement mills. Southeastern Texas and southern Louisiana contain heavy beds of gypsum and anhydrite, particularly as cap rocks to the salt domes of that region. As a rule, they are too deeply buried to be mined now.

The resources of the Rocky Mountain States are too numerous to be set forth in detail; note will be made only of those areas that possess



commercial importance, actual or potential. As a rule the deposits are remote from large centers of consumption and necessarily have been worked only in a small way to supply local needs. In Montana gypsum is most abundant in the Carboniferous of the eastern mountain ranges, and has been worked in Fergus, Carbon and Cascade Counties. The deposits on the flanks of the Big Snowy Mountains, notably, are extensive and of high grade. Rock for Portland cement manufacture has been obtained in Jefferson County. Madison and Big Horn are also to be listed among the counties that have supplies.

Wyoming is endowed with large, but so far meagerly exploited resources distributed in beltlike outcrops over many of the northern, central and eastern counties. Rock gypsum and gypsite have been obtained in the Laramie Mountains, Albany County, principally around Laramie and Red Buttes. Near Greybull and Stucco, Big Horn County, are openings in rock gypsum that belong to the Chugwater formation of Permian age. This formation is represented over wide areas in northern and central Wyoming, usually with one or more gypsum beds. The Black Hills of Wyoming and South Dakota has a surrounding belt of gypsum-bearing beds, known as the Spearfish formation, which is regarded as Triassic in age. The Wyoming section is largely unexplored but in South Dakota operations have been carried on at Hot Springs, Rapid City, Blackwater, Piedmont, and other places as demand warranted. The gypsum occurs in several beds from a few feet to 20 ft. thick or more, and certain layers are almost pure.

In Colorado the deposits of most interest industrially occur along the eastern Rockies, from Larimer County in the north to Custer and Huerfano in the south. They are mainly of Carboniferous time. In Larimer County, Loveland has been a quarry center. Near Colorado City and other places in El Paso County the deposits attain a thickness of 60 ft. or more. Much gypsum rock for Portland cement has been obtained from Stone City, Pueblo County, and from Coaldale, Fremont County. Calcined plasters have been manufactured on a relatively small scale for local markets.

New Mexico has many thousands of square miles underlain with rock gypsum, mainly in the central and southeastern parts. Only the Pecos River belt, which enters the state from Culberson County, Texas, and extends northward for 200 miles or more past Carlsbad and Roswell, has claimed much attention industrially, and there it is the abundant gypsite that overlies the rock that has been mainly worked. Remoteness from important consuming centers limits the use of the resources. The dune deposits of the Tularosa desert, near Alamogordo, illustrate the lavish hand with which nature has scattered the supplies in this region.

The principal resources of Arizona appear to be those in Cochise, Pima and Pinal Counties in the southeast, Navajo County in the east-central part and Mohave County in the northwest, bordering Utah and Nevada, but very likely there are substantial outcrops elsewhere. Activity has been directed mainly to the working of gypsite for wall plaster. The principal operations have been around Douglas, Cochise County.

Central Utah, south of Utah Lake, contains some notable exposures, one of which at Nephi, Juab County, is stated by R. W. Stone<sup>12</sup> to show a vertical face of 400 ft., thickened as it is by folding. The greater part of the deposit, 250 to 300 ft., is high-grade rock and has been used for Keene's cement, hard wall, casting and finishing plasters, for which the market includes the territory west to the Pacific coast. San Pete, Sevier, Millard, Iron, Kane, Washington, Wayne and Garfield Counties have deposits, most of which are still unprospected. Notable are the huge crystals of selenite obtained from caves in the southern part of the state. The San Rafael swell or dome of Emery County, according to the article by Lupton contained in Stone's work,<sup>12</sup> carries gypsum strata on the western flank, for 60 miles, with individual seams 50 ft. thick.

Gypsum has been mined around Lovelock, Carson, Ludwig and other places in western Nevada, for local and coastal trade. In southeastern Nevada, on the lines of the Los Angeles and Salt Lake railroad, supplies have been obtained for the markets of southern California. The rock in the western section belongs to the Triassic, that of southeastern Nevada mostly to Carboniferous.

California obtains a part of its requirements of gypsum, as already noted, from Nevada, but has deposits of its own, largely gypsite, serviceable mostly for the common grades of calcined plaster and for fertilizer. The occurrences of this material are in the Coast Range, in the stretch from San Benito to Los Angeles County. Rock gypsum, said to be in extensive beds of high quality, is found in the Palen and Maria Mountains, Riverside County, but transportation is a drawback to development. Gypsum also occurs as thin layers formed by the drying up of periodic lakes. Northern California seems to lack in important deposits.

Oregon has gypsum supplies in the Snake River country on the eastern border, where mining operations have been intermittently conducted as a basis for manufacture of calcined plaster. There are occurrences, also, in Cook, Grant, Jackson, Josephine and Wheeler Counties, but little is known as to their commercial possibilities.

Washington, lacking developed resources, has imported rock gypsum from Alaska for manufacture. The deposits there are found on the eastern side of Chicagoff Island, near Iyoukeen Cove. The gypsum has been mined to considerable depth and the presence of anhydrite is reported.

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<sup>12</sup>References are at the end of the chapter.

## CANADA

Gypsum deposits are distributed over many provinces, but especially important for industry are those of Nova Scotia, New Brunswick and Ontario, tributary to the eastern markets. Nova Scotia, by reason of the magnitude and the accessibility of its resources for water shipment, ranks first in production; in fact, contributes the major share of the Canadian output. The gypsum of that province occurs in heavy beds, occasionally 100 ft. thick, or more, in association with limestones and shales of Carboniferous age. Anhydrite occurs as included bodies, also in separate beds that by faulting may be brought into juxtaposition with the hydrated rock. There is little doubt that originally all the calcium sulphate was in anhydrous form. The outcrops of white gypsum and anhydrite are prominent features on the shores of Bras d'Or Lake and the northern coast of Cape Breton Island. On the mainland gypsum occurs in Antigonish, Pictou, Cumberland, Victoria, Inverness, Colchester, and Hants Counties, in extensive areas. The important centers of production have been Cheticamp, Aspy Bay, Walton, Cheverie and Windsor, of which the last three are in Hants County on the shore of Minas Basin, an arm of the Bay of Fundy. Open quarry work is the usual method of extraction and costs of production are low, much below the costs prevailing in most of the eastern districts of the United States. The rock is gray, mottled or white, mostly high grade and some of alabaster purity. Much of the quarry output is shipped crude to the United States for manufacture at seaboard plants into wall plasters, board and block.

New Brunswick contains numerous occurrences of gypsum and associated anhydrite, of which the principal ones commercially are in the tidewater district of Chignecto Bay and the Petitcodiac River, Albert County. The beds are smaller in size than those of Nova Scotia, so that underground mining is sometimes necessary, but they afford some exceptionally pure rock. Hillsborough is the chief mining center.

The productive district of Ontario is in an area west of the Niagara River and north of Lake Erie along the Grand River Valley. The gypsum occurs in the Salina formation, which extends eastward into New York. It is mined for Portland cement plants and for calcined plasters, which are marketed in southern and eastern Canada. In northern Ontario gypsum outcrops in force on the Moose River and its tributaries in the James Bay region, but is undeveloped. Quebec possesses considerable showings on the Magdalen Islands, Gulf of St. Lawrence, where the gypsum is found in Carboniferous strata, similar to its occurrence in Nova Scotia.

Manitoba has deposits of economic interest on the lake of that name, 170 miles northwest of Winnipeg. The gypsum has supplied some of the needs of the province. Alberta is known to have extensive deposits near Peace River and in other districts, but they are undeveloped. The

same is true of Saskatchewan and the Northwest Territories. British Columbia has deposits of gypsum in the interior, which for that reason are not readily available. Mining in a small way has been carried on near Falkland, Kamloops district and at Spatsum, in the Ashcroft district.

#### OTHER COUNTRIES

Considerable gypsum is mined in Lower California, Mexico, for export into the western United States. Otherwise little is known about the gypsum resources of Mexico in particular, although from a general standpoint there can be little doubt that they are extensive. Geological conditions similar to those in southern Arizona and New Mexico, with their plentiful supplies, probably prevail across the line in Sonora and Chihuahua. Farther south, Durango and Nuevo Leon are reported to have beds associated with Cretaceous strata. Deposits are also known in the State of Guerrero, south of Mexico City. The gypsum industry is small and little use is made of gypsum for building purposes, aside from calcined plasters.

The island of Cuba is known to contain important deposits, some of which are near Matanzas, where manufacturing of gypsum plasters has been carried on, and near Punta Alegre, east of Caibarien. The latter deposits, which outcrop in a series of hills that fronts directly on the coast, have been worked principally for cement retarder. Gypsum for such use is also obtained at Mariel.

In Argentina large bodies are reported to be included in Mesozoic strata of the Cordillera Range in the provinces of San Juan, Mendoza and Neuquen, but they are not actively worked. Of more importance for industry are the beds in Hernandarias and Curtiembre, Province of Entre Rios.

Great Britain is a leading producer of crude and manufactured gypsum. The deposits of most importance are associated with the Triassic beds of the northern and middle counties of Cumberland, Nottingham, Stafford, Derby and Westmoreland. Calcining plants are operated at Carlisle, Nottingham, Newark and New Biggin, London and Liverpool. The two last named are being supplied with gypsum imported from Nova Scotia.

France, with its famous Paris basin, takes high rank in the industry, especially in the manufacture of calcined plasters which are the basis of an important foreign trade. The beds, which are of Tertiary age, attain a thickness of 50 to 60 ft., and occur at several horizons in the series of clay strata. The gypsum of the Paris basin carries 10 per cent or so of lime carbonate. Outside of that area gypsum is found in Savoie, Haute Saône, Jura, Saône et Loire, Charentes and other departments. There are reported to be 200 or more quarries and manufacturing plants in the country.

Germany has a large gypsum industry, based on extensive deposits and a well developed technology. It also has a substantial export trade. The deposits are found in the Harz Mountains of central Germany, in Thuringia, Saxony, Hesse, Bavaria and other parts. The Permian beds of the Harz region are most important. Germany uses large quantities of calcium sulphate for the recovery of sulphuric acid.

Spain is able to satisfy its requirements of gypsum from deposits in Catalonia, Aragon and Andalusia. Italy is well supplied with gypsum, particularly in Piedmont and Tuscany, where it occurs in beds of Triassic age. The sulphur deposits of Sicily are accompanied by anhydrite and gypsum.

Russia, with its varied geological structure, has abundant resources distributed among formations that range from the Devonian to the Tertiary. Its gypsum industry is believed to be very important, although details are lacking. That gypsum is abundantly distributed over many parts of the Asiatic continent is indicated by its occurrence in those countries that have been more thoroughly explored. Turkey, Syria, Persia, Turkestan, India and China are among the number known to contain deposits of commercial value.

Egypt has a historic relation to the gypsum industry by reason of the use of calcined plasters in building in very early times, and is counted among the substantial producers of structural gypsum of the present day. The Union of South Africa contains deposits that yield rock for Portland cement manufacture and calcined plasters.

In Australia, all states have gypsum deposits, but those in South Australia are the most important.

#### POLITICAL AND COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

As deposits of gypsum are so well distributed and of such ample size, most countries that rank as important consumers are able to obtain the major part, at least, of their requirements from local sources; therefore gypsum does not figure largely in international trade and rarely enters into consideration for control of its movement by tariffs, at least in the crude form. Examples of large-scale shipments of gypsum from one country to another at present are found in the exports from the Maritime Provinces of Canada to the eastern seaboard of the United States, which exacts no duty on the raw, unground product, and to England under reciprocal tariff agreement. The difference between low-cost ocean freights and higher rates for rail transportation makes such exports feasible. The imports have reached the important total of a million tons in periods of active demand, representing the greater share of the world trade in rock gypsum.

Duties, however, are often levied upon the ground or calcined products of gypsum as a protective measure. The rates currently in force in the United States are as follows:

MATERIAL	DUTY PER TON
Crude, unground gypsum . . . . .	Free
Ground gypsum . . . . .	\$ 1 40
Plaster of Paris . . . . .	1 40
Keene's cement, or other gypsum valued at \$14 or less per ton . . . . .	3 50
Over \$14, not over \$20, per ton . . . . .	5.00
Over \$20, not over \$40, per ton . . . . .	10.00
Over \$40 per ton . . . . .	14 00
Manufactures of plaster of Paris 35 per cent ad valorem	

Canada levies no import duties on the crude rock, but has the following rates for manufactured or calcined gypsum: For calcined plaster and prepared wall plasters the general duty is 12½¢ per 100 lb., with a British preferential rate of 8¢; for ground gypsum, not calcined, the rate is 15 per cent ad valorem, general and 10 per cent preferential.

Among European countries Great Britain, France and Germany rank first as exporters of gypsum and its products. With the exception of some countries that depend altogether upon foreign sources of supply, the movement is small. Among the principal importing countries are Norway, Denmark, Sweden, Holland, Belgium and Czechoslovakia. In not a few countries there is as yet little demand for gypsum structural materials, and consequently small use is made of the native resources even when these are believed to be abundant.

Few authentic records of the early history of gypsum mining and manufacturing in this country are available or accessible today. The industry had a slow, uneventful development for the first years, restricted as it was to the supply of crude gypsum for grinding into land plaster for the needs of agriculture. Equipment and methods for producing this material were simple and for the most part inexpensive. The manufacture of calcined plasters from native rock was not introduced widely until the latter part of the nineteenth century.

As far as the available information goes, it would appear that gypsum was first obtained on a commercial basis in Onondaga and Madison Counties, New York, as a consequence more or less of the embargo placed upon importations of foreign gypsum early in the last century. For some time before 1800, gypsum in crude state was brought from Nova Scotia and also probably from France for grinding into land plaster. The original discovery of the gypsum deposits of central New York seems to date back to 1792, in which year William Lindsay is on record as finding the mineral in the town of Camillus, Onondaga County. A decade or two later the deposits of that region were regularly exploited, as noted in the local histories of J. V. H. Clarke<sup>3</sup> and J. E. Smith.<sup>9</sup>

Appropriately, the name Camillus has been applied to the geological formation that carries the gypsum and rock salt in New York.

Production of gypsum in the district was stimulated by the opening of the Erie Canal (1825), which permitted shipments westward to the Great Lakes and eastward to the Atlantic seaboard. For many years this area contributed most of the domestic product which, however, the did not reach as much as 100,000 tons until well after the middle of nineteenth century.

Manufacture of calcined plaster began in the Hudson River region about 1835, but local gypsum, it appears, was not used for the purpose. Rapid expansion in the uses of calcined plasters for building construction may be noted in the years following 1890. By that time, calcining plants had been set up in Michigan, Iowa, Kansas and other states. In that year for the first time the amount of crude gypsum converted into stucco, according to *Mineral Resources* of the U. S. Geological Survey, exceeded 100,000 short tons out of a total mine product of 182,995 short tons. The land-plaster output, in comparison, was 65,525 tons. By 1900 the output of the mines and quarries in the country had grown to 594,462 tons and by 1910 to 2,379,970 tons. Wide-scale expansion in the demand for structural gypsum materials was mainly responsible for this remarkable growth of the industry, although the advance of Portland cement manufacture accompanied by an increased use of raw gypsum as retarder played some part. The peak of growth was reached in the years from 1923 to 1929, with an average output in excess of 5,000,000 tons crude product. Building operations were sharply curtailed in the next few years, so that activity fell away to a fraction of the former rate, and the industry has not since regained its former importance. The gypsum output in 1935 was about 1,937,500 short tons and the total for calcined plasters was about 1,200,000 short tons. The sales of domestic gypsum and its products in that year were valued at \$18,860,348, distributed among many items, of which the largest was wallboard, worth \$5,515,944.

The foreign trade in gypsum and its products at present has a substantial balance on the side of imports as against exports. This condition has obtained for a number of years and mainly results from the entry of crude gypsum from Canada along the Atlantic seaboard for manufacture and sale to the eastern trade. Some crude gypsum is shipped from Mexico to the Pacific coast. The importations of rock have amounted to about 25 per cent of the total mined in the United States; for 1935 the figure of imports was 450,250 short tons, which in the forms sold to consumers was valued at \$4,826,797. In former years the imports have run to much larger figures, although the proportions of foreign to domestic gypsum in the trade have not shown much change.

The exports of gypsum were once much more of a factor in the industry than at present. From about 1920 to 1929 foreign demand for wallboard and plasterboard, particularly, was fairly keen, but the sales fell

off rapidly after that period. In 1934 the exports of gypsum, crude and manufactured, were valued only at \$133,492, but increased to \$186,196 in 1935. The world production of gypsum is not a matter of exact determination, since reliable statistics for some countries are unavailable. The most complete table, probably, is that published in the Minerals Yearbook of the U. S. Bureau of Mines. According to this authority the world output in 1934 was 7,800,000 metric tons, with estimates only for a number of countries and for some, like Mexico and the Union of Socialist Soviet Republics no figures are apparently included.

### TECHNOLOGY OF ITS PRODUCTION AND ITS USES

BY H. J. BROWN

The spectacular growth in the consumption of gypsum and gypsum products in the United States since the turn of the century may be attributed wholly to the expansion in building. During this period more rapid methods of construction have been required, and a general awakening to the importance of fire resistance and fire-retarding materials has greatly increased demand for gypsum products. By inherent qualities of rapid setting and hardening, gypsum products, especially wall plasters, are particularly suited to improved and more rapid methods of construction, and the resistance to attack by fire has helped to meet the increased demand for fireproof construction. Manufacturers, alive to the opportunities for expanding business, have overcome defects in material and, through research, developed improved technique in manufacture and application. New products, such as gypsum block, wallboard and plasterboard, and other specialties, have been placed on the market, contributing greatly to increased output.

Many combinations of companies into single organizations have been effected. A number of new plants have been built; some because of obsolescence of older ones, and others for entirely new operations in localities not previously exploited. Although there have been failures to survive because of the faulty selection of deposits and inability to meet competition by reason of location, the present surviving units of the industry certainly give promise of continued operation. The units that have survived the lean years of depression should profit by any expansion in building during the next decade.

There are some 64 plants producing gypsum products within the United States. The majority of these consume domestic rock, but about 10 per cent operate on imported materials. The aggregate over-all capacity in terms of raw material exceeds 25,000 net tons per day—an amount that apparently is sufficient to meet any reasonable expansion.

### MINING METHODS

Gypsum being a material of low value in the crude state, only deposits that may be exploited at low cost are of economic importance. The



quarrying or mining methods for gypsum or gypsite are essentially the same as for many of the nonmetallic minerals. Quarrying is usually resorted to where the depth of overburden is not excessive in comparison with the volume of the gypsum to be recovered. Quarry methods follow in general the same practice as for limestone. Where the cost of recovery of the gypsum by quarrying is prohibitive because of the type and thickness of overburden, mining is done through shafts, slopes or adit tunnels. The method followed depends upon the character of the individual deposit. In flat-lying or slightly pitching seams the usual practice is that of room-and-pillar mining. Occasionally, where the seam or deposit pitches at an angle too high to permit of this method, either overhand or underhand stopping is undertaken. In the quarrying and mining operations, hand work is common, but some are partly or even completely mechanized. Capacity, capital investment and costs of extraction are the determining factors.

Gypsite, because of its physical characteristics, is usually won by the simple process of shoveling off, or removing by scraper or power shovel, a thin covering of overlying clay or soil and then loading the gypsite by hand, scraper, shovel, drag, or excavator line, for transport to the mill or processing plant.

By-product gypsum obtained from the waste of chemical plants manufacturing phosphate products is an occasional source of raw material, but it does not constitute a large proportion of that used in gypsum manufacturing. This by-product gypsum is often delivered to the gypsum plants by pipe line in the form of a sludge, or it is dug from waste piles or ponds surrounding the phosphate plants, and must be dewatered and partly dried by centrifugal filters or presses before it can be used.

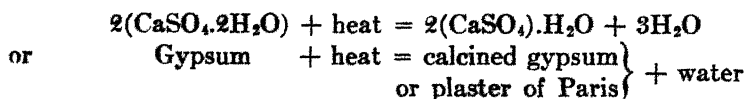
Rock gypsum is the only raw material from which all gypsum products can be made. Gypsite is principally used in the manufacture of wall plasters, in which its inherent plasticity, brought about by fine division of clay and organic admixtures, is unsurpassed for ease and speed in application. By-product gypsum closely approaches rock gypsum, in that nearly all gypsum products can be made from it by proper processing, and its products successfully compete with those from the rock.

#### USES AND PREPARATION

Table 1 shows the classification of raw gypsum, its products and their uses. In the manufacture of these products, three steps are involved: crushing, grinding and calcining. The crushing and grinding, and also the screening, conveying, elevating and transporting involved in the processing, are done with machinery and equipment similar to, and often identical with, that used in other nonmetallic industries, particularly that used in manufacturing Portland cement.

† Calcining, which involves removal of part, or at times all, of the water of crystallization from the gypsum, is done by heating, either in kettles

or rotary kilns. Pure gypsum contains 79.1 per cent of calcium sulphate and 20.9 per cent water of crystallization and is expressed chemically as  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , but, in the process of calcining the water of crystallization is reduced to 14.7 per cent, leaving a calcined product with the chemical formula  $2(\text{CaSO}_4) \cdot \text{H}_2\text{O}$  or, as the half-hydrate is frequently written,  $\text{CaSO}_4 \frac{1}{2} \text{H}_2\text{O}$ , with 93.8 per cent calcium sulphate and but 6.2 per cent of water. The chemical reaction for calcining of gypsum is:



Although calcining may be carried on in rotary kilns as well as in kettles, production from the latter accounts for some 80 per cent of all calcined output. The kettles are the only type of machinery peculiar to the gypsum industry. The conventional type consists of a cylindrical shell some 10 ft. or more in diameter, with transverse flues, holding from 12 to 14 tons or even more raw material. This shell is set in brickwork surrounded by a steel jacket; it is fired underneath by hand or by stoker with coal, oil or gas, and fed by conveyer from overhead. Kettles are discharged when calcining is complete into a "hot pit," from which the product is elevated and conveyed to bins for storage or further processing or treatment.

Depending upon the fineness to which the crushed gypsum is ground, its purity, and the efficiency of the kettle installation, the calcining process takes approximately two hours, on the average. In the most efficient plants one hour suffices. Ground gypsum calcined to the half-

TABLE 1.—*Raw Gypsum and Its Products*

Gypsum Rock (Base Material)	{ For export; for use in the manufacturing of other gypsum products; as a flux in the smelting of nickel ores; in the "Burtonizing" of brewery water; as alabaster, for carvings, statuary and <i>objets d'art</i> ; Keene's cement and similar products.
1. Crushed Gypsum	{ Used as a retarder in the manufacture of Portland cement and in the manufacture of other gypsum products.
2. Ground Gypsum	{ Used as land plaster in the conservation of moisture in the soil and for nitrogen absorption from manures; diluent for other fertilizer compounds; as a filler in paint, paper, cloth, etc.; in insecticides; as terra alba; in manufacturing calcium sulphide, ammonium sulphate and sulphuric acid; in the manufacture of Keene's cement.
3. Calcined Gypsum	{ Used in plasters for interior walls; plaster of Paris, molding, casting and finishing plasters; dental and surgical plasters; plaster for bedding plate glass while grinding and polishing; pottery-mold plaster; water hardener for brewery water; dehydration of oil; filtering, etc., and for the manufacture of special products.
	{ For manufacture of partition block, tile and forms; for plaster-board; wallboard, insulating board, etc.; for structural roof and floor decks and "poured or erected fireproofing."

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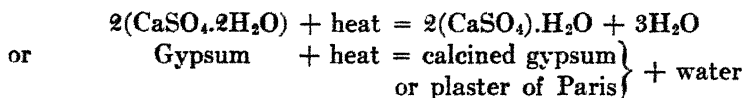
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hydrate requires approximately 60 or 70 lb. of coal of fair quality, or the equivalent of this fuel in either oil or gas, to the ton of finished product. The temperature at which the material is considered calcined varies with the use to which the finished product is put, and may range from 300° to 350° F. for the usual output of calcined gypsum. It may be carried, however, as high as 400° F. for special products; such, for example, as those supplied to the plate-glass industry for use in bedding glass for grinding and polishing.

Modern kettles are equipped with both registering and recording thermometers as well as other safeguards to ensure proper control of calcining temperatures and length of calcining time. Great care is taken to ensure uniformity of product and frequent and practical testing is pursued to ensure uniform quality and time of set. Particular care is taken to ensure freedom from contamination from raw gypsum particles, as such dilution, even in minute quantities, affects the strength and time of set of the product.

For calcining in rotary kilns, neither grinding prior to calcining nor drying preparatory to grinding is necessary. Kiln feed is usually all under 1½ in. in diameter. For the most uniform calcining it is essential that the fines be removed, since the rate of heat transfer from the surface to the center of the fed material progresses at a different rate with the size of varying particles—the output of the kiln would not be calcined uniformly with a feed varying greatly in particle size. Kettle calcining, being a batch process, can be controlled with great exactness, and the product is suitable for all common purposes for which calcined gypsum is used. The product of the rotary kiln is most adaptable for use in wall plasters, where extreme exactness in time of set is not required or can be controlled during mixing, or for use by the manufacturer in his own plant for making block and board.

While the water content as shown by analysis may indicate that the rotary-kiln product is a material similar in all respects to that calcined by the kettle process, microscopic inspection reveals a mechanical mixture of raw gypsum associated with material in different stages of calcination.

In overcoming this drawback to the rotary kiln as a calciner, it has become good practice to crush the hot kiln output directly as it comes from the kiln and then immediately to grind in tube mills to the finished fineness desired. By this step the material is thoroughly blended and the calcining process continued on such parts of particles as are still undercalcined or raw, owing to the residual heat combined with the heat that is developed in grinding. In addition, particles that have been burned or overcalcined are partly rehydrated with water driven off by the continued calcining process, resulting in a final product much more uniform in composition and with characteristics more comparable with kettle-calcined material.

Producers of large output have generally combined kettles with the rotary kiln so that the fines rejected in the kiln process, which they might not be able to dispose of as an intermediate product or to process further successfully, might be used to advantage in the production of any calcined product.

Arrangement of apparatus in a plant varies in keeping with individual experience and trade demands. Except for maintenance, supervisory labor only is required. The heaviest labor charge is for mixing, packing, warehousing and the loading of the finished product.

The greatest tonnage of gypsum products is sold calcined—mixed with fiber, retarder and often sand—as wall plaster. In the manufacturing of this commodity it is now quite usual to further grind the calcined material in tube mills to varying degrees of fineness, for the purpose of still further increasing plasticity or workability, after the suggestion originally made by Emley, of the United States Bureau of Standards. Even gypsite plasters yield increased plasticity by undergoing this treatment. Other large tonnages are also converted, usually by the original manufactures, into gypsum block by the simple process of mixing with fiber and water and casting into shape either by hand or by machine, followed by subsequent drying for removal of excess mixing water.

Gypsum wallboard, plasterboard and lath are similarly made by continuous casting of calcined gypsum mixed with water and other ingredients between two continuously moving sheets of special paper, the combination being pressed to shape and uniform thickness and then cut off to the required length while moving. Such boards are dried as they come from the machine, without further handling before that necessary for warehousing and shipping.

Special products, in addition to those mentioned above, are made by some manufacturers, and although they do not comprise an important tonnage they command attractive prices. Such products are represented by terra alba and special fillers, and the dental and surgical plasters. The sole exception to this is in the manufacture of Keene's cement, a special product of individual characteristics. One manufacturer is generally credited with the bulk of production. Centrally located in the West, the output finds markets throughout the country: a proceeding unusual in the industry, in which shipping radius is somewhat restricted by freight rates to center of consumption.

#### TESTS AND SPECIFICATIONS

The tests for gypsum and gypsum products are both chemical and physical. They vary in complexity from the rapid checks used in plant control to those used for investigation and in scientific research.\*

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\* Committee C-11 on Gypsum, American Society for Testing Materials, has sponsored and published as Standard Methods of Testing Gypsum and Gypsum Products A.S.T.M. designation C-26-33, which may be found in the volume on A.S.T.M. Standards for 1933.

In the quarry and mine usual inspection of the output by experienced operators, with periodic chemical or microscopic analysis, ordinarily suffices to prevent dilution with dirt, anhydrite, or with roof or bottom rock. Because of the difference in specific gravity and hardness, the experienced operator can readily differentiate between gypsum and anhydrite. In making bulk shipments by rail or water, samples of shipment are reported as to average chemical analysis. Where anhydrite dilution or limitation is important, microscopic analysis involving a proportionate count of the different particles gives reasonably accurate results as to composition. A similar requirement may be necessary in shipment of ground gypsum, except that color comparison may be an additional stipulation when the material is to be used as a filler.

Several tests are made for calcined gypsum. First, the uncalcined but ground gypsum constituting the kettle feed is screen-tested for fineness, not only as a check upon the grinding and separating efficiency but to ensure uniform and more regular kettle duty, for, given equal time and temperature, uniform fineness of raw material makes for shorter time in calcining, conserves power, and helps to assure uniformity in time of set, consistency and plasticity in the product. Next, the time of set is checked frequently to ensure proper calcining and freedom from contamination with uncalcined material, which acts as a very powerful accelerator. Time of set is taken by means of the Vicat needle and the procedure follows closely the method used in testing Portland cement. Consistency—the relationship between a specified amount of plaster and the amount of mixing water required to reach a given fluidity, is also measured by the Vicat needle. Tests for workability of calcined gypsum and plaster made therefrom are made frequently upon the Emley plasticimeter,\* used in the lime-manufacturing industry to determine the plasticity of lime for putty and mortars.

Bending or flexure tests, tests for compressive strength and for water absorption, are made for gypsum block, wallboard and plasterboard.

Except for the very broad specifications for crude gypsum and gypsum products that the industry has attempted to develop for universal application through the American Society for Testing Materials,† it is not usual either to purchase or to sell gypsum products under standard specifications. Each buyer purchases material to suit his needs or methods of operation. In transactions involving crude or crushed gypsum the degree of purity and chemical analysis may be specified and a previously submitted sample is usually the governing check. For use in the manufacture of Portland cement the  $\text{SO}_2$  content is often specified,

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\* See Standard Specification for Hydrated Lime for Structure Purposes, A.S.T.M. designation CC-31, 27, American Society for Testing Materials Standards, 1933.

† See Standard and Tentative Specifications for Gypsum and Gypsum Products. American Society for Testing Materials.

although by no means an infallible guide to desirability. In calcined gypsum, plaster of Paris, molding, casting, and other similar products, fineness, time of set and consistency are of paramount importance. The relative whiteness of the product is often specified where the material is to be used for decorative purposes. In the sale or purchase of wall plaster, time and uniformity of set, plasticity and sand-carrying capacity are the features sought. Seldom is any specification other than time of set made a part of a transaction. Such plasters as a rule are made to conform to the particular kind of work in which they are to be used, or to the particular type of sand or aggregate, and sometimes, even to the water with which they are to be mixed.

Gypsum block, gypsum wallboard and plasterboard, and many of the other gypsum products are more or less standard as to size, thickness and weight, and are commodities purchasable in the open market.\* Often special products are made to meet specific needs; in such cases specifications are a matter for mutual agreement.

#### MARKETING

In common with other staple commodities entering the building industry, gypsum products are sold to the owner, builder or contractor through the building-supply dealers and are stored and delivered to the point of consumption either by such dealer or by the manufacturer from his own warehouse, or plant, if within delivery distance. Being relatively low-priced commodities, shipping and warehousing cost constitute a large portion of consumer's cost, and plants located nearest to consuming centers profit from stable prices. With the industry's capacity exceeding consumption, as it does, competition within the industry and with other competing materials is high. Sales of gypsum products have been extended to all types of construction.

The items entering into the cost of gypsum products to the consumer consist of: (1) the cost of the raw material, whether from domestic mines or quarries or imported; (2) cost of manufacturing into the specific commodities under consideration, into which enter the prices of fuel and other supplies of a primary or secondary nature, such as paper used in the manufacture of board; (3) transportation, warehousing and delivery to actual point of consumption, which probably affects the profit more than any other of the charges.

While selling price in the early history of the industry was based on manufacturing cost at the mill, and varied to the consumer with the amount of freight charged by the railroads for delivery, the custom has grown to accept certain specified mill base prices as applying to given

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\* The National Board of Fire Underwriters has specifications for a number of gypsum commodities and issues special specifications relating to fire resistance, which may be obtained through the Underwriters Laboratories, Chicago, Illinois.



consuming territory. Thus, those plants closest to the market stand to profit most, and more remote plants must either absorb the differential or refrain from selling in the same market except below cost of production.

Overcapacity and an eagerness to keep all plants operating in the face of dwindling markets at times affect prices severely. A satisfactory future for the industry would appear to rest upon development of increased consumption in new building or in other fields, without material increase in plants over present potential capacity. That greater use in the building industry will take place through a more thorough appreciation of the merits inherent in gypsum products is inevitable. Present capacity will be outgrown, greater output will make for lowered costs, and these, in turn, will make for still wider distribution.

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## CHAPTER XX

### HEAT AND SOUND INSULATORS\*

By J. E. LAMAR,† MEMBER A.I.M.E. AND CHARLES F. FRYLING‡

**I**NSULATING materials include a wide variety of nonmetallic mineral products such as exfoliated vermiculite, expanded gypsum, 85 per cent magnesia, diatomite, asbestos and mineral wool. Except mineral wool, these products, or the materials from which they are manufactured, are described in other chapters of this book.

#### PROPERTIES OF INSULATING MATERIALS

The insulation properties of the materials considered in this chapter result in general from entrapping extremely small pockets of air in such a manner that heat is not transferred by convection. The minimum heat conductivities of such insulating materials are therefore slightly greater than the conductivity of still air. In addition, conductivity varies with the over-all density of the material, the temperature range in which the measurements are made, and the form into which the material is fabricated. However, the numerical value of heat conductivity should not be the sole criterion of an insulating material: for a particular application it may be advantageous to use a material of somewhat higher conductivity in order to secure other desirable engineering properties. This is especially true with certain materials that are used because of their high refractoriness.

The comparative insulating value of certain insulating materials is shown in Table 1. In comparing the values presented in this table, it should be remembered that the heat conductivity varies with both the temperature range and over-all density. For a graphic treatment of these factors as they apply to mineral wool, the reader is referred to the article by J. A. Schaeffer.<sup>29</sup>

Sound-insulating materials<sup>1,17,22</sup> serve two purposes, to absorb sound and to reduce its transmission. Sound-absorbing products are used to

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‡ Chemist, Non-fuels Division, Geochemical Section, Illinois State Geological Survey.

<sup>29</sup> References are at the end of the chapter.

improve the acoustics of theaters, churches, etc., and usually are employed in the form of porous tile, block or plaster. Unlike heat-insulating materials, which depend for their insulating properties on the presence of numerous small air pockets, sound-absorbing materials owe their properties to the existence of continuous channels with which are con-

TABLE 1.—*Heat Conductivity of Various Insulating Materials*  
British thermal unit feet per hour per square foot per degree Fahrenheit<sup>a</sup>

Material	Density, Lb. per Cu Ft	Temperature, Deg. F.	Heat Con- ductivity
Diatomite, in powder form <sup>b</sup> . . . . .	10 6	86	0.026
Diatomaceous earth bricks <sup>c</sup> . . . . .	27.7	1600	0.077
Spun glass blanket <sup>c</sup> . . . . .	5.56	400	0.043
85 per cent magnesia pipe covering <sup>c</sup>	14.5	400	0.046
Bauxite molded with clay <sup>c</sup> . . . . .	33.0	1600	0.100
Expanded vermiculite <sup>b</sup> . . . . .	8 0	100	0.04
Rock cork, slabs, asphalt binder <sup>c</sup>	15 6	86	0.027
Rock-wool filling, loose <sup>c</sup> . . . . .	10 0	90	0.023
Rock wool, felted, blankets <sup>c</sup> . . . . .	8 2	300	0.034
Rock wool, felted, with metal jackets <sup>c</sup> . . . . .	19 4	400	0.044
Rock-wool cement <sup>c</sup> . . . . .	30 0	500	0.085
Slag wool <sup>d</sup> . . . . .	12.0	85	0.02-0.025
Still air <sup>c</sup> . . . . .	0.08	32	0.013

<sup>a</sup> Should be multiplied by 12 to convert to B.t.u. inches.

<sup>b</sup> J. A. Schaeffer: reference 29. Recalculated.

<sup>c</sup> *Chem. and Met. Eng.* (1932) 39, 658. Recalculated.

<sup>d</sup> L. S. Marks: *Mech. Engrs. Handbook*, Ed. 2 (1924). Recalculated.

nected many small pores. The sound waves are admitted to the insulating material by the channels and are trapped by the pores, where they are ultimately dissipated in the form of heat.

The reduction of the amount of sound transmitted through walls, partitions, etc. is accomplished principally by reflecting the sound by means of dense, smooth-surfaced tile, block or plaster. Sound-absorbing materials may be used also to aid the process by reducing the incident sound.

Usually the manner of fabrication of the various nonmetallic minerals used for sound insulation is more important than the character of the raw material itself. As a general rule, however, it may be said that fibrous or cellular nonmetallic minerals that are good heat insulators can be converted by proper methods of manufacture to products of value for sound insulation.

Data available regarding the efficiency of nonmetallic minerals as sound insulators refer chiefly to the diverse products in which they are used, and are of little importance from the standpoint of raw materials because the method of manufacture largely influences their properties.

## INSULATING MATERIALS

*Asbestos*.—The term “asbestos” is used commercially to describe any mineral that can be separated readily into more or less flexible fibers (ref. 18, p. 43 and ref. 6). All the commercial varieties of asbestos are either amphibole or chrysotile, the latter being the most useful variety.<sup>6</sup> As an insulating material the usefulness of asbestos depends to a considerable degree on its incombustibility and on its fibrous structure and the flexibility of the fibers, which permits it to be woven into fabrics (ref. 18, p. 62). (See chapter III.)

Asbestos is used for heat insulation as crude asbestos, asbestos sand and fiber, and in various manufactured forms such as yarn, cordage, cloth, paper, boards and blocks. Asbestos cements made of short-fiber asbestos and clay or other binder are used extensively as pipe and boiler coverings (ref. 6, p. 8 and ref. 27, pp. 129–130). Asbestos felt, cloth and plaster containing asbestos are used for acoustic work.<sup>27</sup>

*Basic Magnesium Carbonate*.—Basic magnesium carbonate (see chapter XXIV) is made chiefly from dolomite, by a chemical process that involves calcination of the dolomite followed by slacking to give calcium and magnesium hydroxides. These hydroxides, when treated with carbon dioxide in the presence of water, yield the relatively insoluble calcium carbonate and the soluble magnesium bicarbonate. The latter is separated from the calcium carbonate by filtration and recovered from solution by boiling, which converts it to the relatively insoluble basic magnesium carbonate (ref. 18, p. 205). This carbonate is used in the manufacture of pipe and boiler coverings for heat insulation. The product “85 per cent magnesia” widely used for pipe covering is composed of about 85 per cent of basic magnesium carbonate and about 15 per cent of asbestos fiber.

*Diatomite*.—Diatomite (see chapter XIII) is used for heat insulation in the form of natural and calcined powders, natural aggregates, and molded, pressed and natural brick.<sup>25</sup> It is also employed for sound deadening in buildings, music rooms, telephone booths, etc.<sup>9</sup> The powdered material is produced by grinding raw or calcined diatomite. The natural brick are sawed from the diatomite deposits. The other types of brick are made either with or without a binder and usually are fired.

*Gypsum*.<sup>8</sup>—Calcined gypsum (see chapter XIX) is used chiefly as an ingredient in insulating plasters and in an expanded form known as “Insulex.” The product is obtainable in either powder or flake form. The powder is calcined gypsum (plaster of Paris) to which certain chemicals that produce gas in the presence of water have been added by the manufacturer. When water is added the material is expanded by the liberated gases and the set of the plaster of Paris is so timed that it occurs when maximum expansion is reached, resulting in a light-weight, cellular

product. The flake form is supplied dry, for use where a wet process of installation is impossible.

*Vermiculite.*<sup>26</sup>—Vermiculite is a mica that expands, when heated to a moderate temperature, into tabular masses composed of numerous thin sheets. (See chapter XXVI.) Zonolite is a trade name applied to vermiculite from the deposit at Libby, Mont. The expanded vermiculite is graded according to size and used either as loose granular material or as blocks, slabs, boards, etc., into which it is fabricated after the addition of a suitable binder. It is used for both heat and sound insulation.

*Mineral Wool.*—Mineral wool is an artificial product composed of extremely thin, silicate fibers, mostly 1 to 10 microns in diameter, and derives its name from its resemblance to sheep's wool. It is widely used for insulation and owes its insulating properties to innumerable, minute air pockets, which are entrapped by the fibers of the wool and prevent the transfer of heat by convection. The term mineral wool includes rock wool, slag wool, glass wool, glass silk, and silicate cotton. In general the names indicate the raw material from which the wool is manufactured.

#### MANUFACTURE OF MINERAL WOOL

The production of the fiber is fundamentally the same for all the various types of mineral wool, and involves subjecting a molten silicate to shearing forces while it is cooling fast enough to prevent crystallization. There are two groups of methods: (1) in one the molten silicate is broken into separate droplets, which are then subjected to shearing forces; (2) in the other a fine thread is drawn continuously from a mass of molten silicate. The processes in the first class involve formation of droplets by a blast of steam or air and projection of the droplets through relatively still air; disintegration by centrifuging on a large rotating disk; and formation of threads by allowing molten droplets to fall from an elevated platform.\* In the second class are methods such as the ancient procedure of shooting arrows which have been touched to molten glass; spinning a thread on a rotating wheel from the molten end of a glass rod; extruding molten silicate from a container and spinning the threads so obtained on a rotating drum; and subjecting a flowing stream of molten glass to a steady blast of steam or air so adjusted that the glass stream is not broken during its elongation.

Only glasses that do not devitrify readily at high temperatures can be spun. Slags and rocks suitable for woolmaking are apparently too corrosive, at the temperatures required, to be melted in glass tanks made with the refractories now available and it is necessary, therefore, to use cupolas.

The commercial process commonly used for making rock wool involves melting rock fragments in a water-jacketed cupola between  $2\frac{1}{2}$  and

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\* M. F. Goudge: Personal communication.

7 ft. in diameter and 10 to 15 ft. high, using coke as a fuel. The size of the rock is generally greater than 2 in. and less than 10 in. Operating temperatures range from 2500° to 2800° F. The molten rock issues from an orifice at the bottom of the cupola and is blown into fine fibers by a jet of high-pressure steam or air. The fibers accumulate in chambers, from which they are removed for further processing. With the fibers are small beads of glass known as "shot," which are droplets of the molten rock that escaped being blown into fibers.

The commercial production of rock wool by melting raw materials in an electric-arc furnace and spinning the molten rock on a high-speed disk has recently been undertaken in Canada.<sup>26</sup>

The process of making slag wool from cooled blast-furnace slag is generally similar to that used for making rock wool. Some slags require the addition of comparatively small amounts of siliceous materials to yield a cupola charge of proper chemical composition. It is reported that slag has been converted to wool while still in the molten state as it comes from the blast furnace, thus avoiding remelting.<sup>16</sup>

In France a different process is said to be followed.<sup>4</sup> Granulated slag is melted in refractory-lined vessels by heat from oil burners. The vessels have perforated floors through which the slag passes as fine threads to a rotating drum sprayed with water to prevent adhesion of the slag threads.

Glass wool or glass silk is a mineral wool generally made from soda-lime glass. It was developed as a war necessity in Germany, and subsequently has been made commercially in England and in the United States. "Silicate cotton" is another term sometimes applied to glass silk. There are various methods of making glass silk, some of which are not as yet fully described in the literature. According to Carlisle:<sup>7</sup>

Ordinary commercial glass is used as raw material for the manufacture of "glass silk" and is heated in electric furnaces which are provided with apertures through which the glass exudes when it reaches the molten state. As it exudes, a skilled operator touches the spot with a glass rod, a strand of the glass is drawn out, and this is deftly whisked across a revolving drum alongside the furnace. The drum continues to revolve rapidly, drawing out strands from all the apertures and lapping them over and over until the drum is covered with a thick felt of thin glass fibers. A cut is then made across the drum, and the spun glass comes away in the form of a blanket of fibers, each in the neighborhood of 0.001 inch in diameter. The fibers, as they come from the drum lie almost parallel and possess such tensile strength that they can be folded and crumpled with no ill effect. Such blankets of fibers are "opened out" into fine layers which have the appearance of silky veils and they are laid one upon another until sufficient thickness has been built up to give a good degree of heat insulation.

A recently developed process consists in subjecting a stream of molten glass to the action of a steam or air blast whose velocity is so adjusted that the molten glass is drawn out into a long, thin, continuous fiber, which is collected in a suitable manner. Though the details of this

operation have not been described, it appears to be well known that one glass wool that finds ready acceptance on the market is made in this manner. The product is lustrous, free from shot, but is inclined to have a somewhat coarse fiber having a diameter roughly between 10 and 25 microns.

Another process<sup>2</sup> not fully described, which may be a modification of the process mentioned above, involves the application of steam, which "brings molten glass to a conveyor line in fluffy, downy wisps somewhat resembling falling snow or down of geese," from which form it is converted into thread or yarn.

#### MATERIALS FOR MAKING MINERAL WOOL

*Glass Wool.*—Little specific information is to be found regarding the composition of the glass used in making glass wool or glass silk. The glass is variously described as "high-quality glass,"<sup>3</sup> "ordinary commercial glass,"<sup>7</sup> and as "soda-lime glass."<sup>11</sup> A determination of the soda content of two glass wools indicates that the percentage of this component is somewhat in excess of that commonly found in soda-lime glass. The raw materials used in making the glass from which wool is manufactured are, as far as is known, similar to those used in making glass for other purposes. The latter are described in standard works on glassmaking. Discussions of sources of raw materials for glass making, especially silica sand, limestone and lime, are to be found in books on economic geology, including publications of federal and state geological surveys.

*Slag Wool.*—Slag wool is made chiefly from the slag of iron blast furnaces, though copper slag has been used in California<sup>21</sup> and lead slag has been used in numerous other places.<sup>10</sup> The cupola charge may or may not require the addition of calcareous, argillaceous or siliceous material, to correct deficiencies in the composition of the slag. A slag containing between 30 and 50 per cent CaO or CaO and MgO would yield a wool without the addition of other substances. The chemical requirements of blast-furnace slag for making slag wool are similar to those for materials for making rock wool, and substances deleterious in the latter are similarly undesirable in slag. Typical analyses of slags and slag wools are given in Table 2.

#### ROCK WOOL

*Rock Wool.*—Rock wool can be made from two general classes of raw materials: "natural woolrocks" and rock mixtures or "composite woolrocks." Both the natural woolrocks and composite woolrocks are composed dominantly of lime, magnesia, alumina, silica and carbon dioxide, although it has been shown that neither alumina nor magnesia is essential.<sup>22</sup> Experiments indicate that in general rocks or rock mixtures containing between 20 and 30 per cent carbon dioxide, or roughly 40 to

65 per cent calcium carbonate or calcium and magnesium carbonates, and the balance chiefly silica and alumina, will make rock wool if properly processed. Table 2 lists typical analyses of woolrocks and rock wools.

TABLE 2.—*Chemical Analyses of Typical Woolrocks, Rock Wools, Slags, and Slag Wools*

	Composition, Per Cent							
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Ignition Loss	S	Miscellaneous
Woolrock: <sup>a</sup>								
Illinois .....	29.76	7.95	2.82	12.21	17.74	27.15		2.37
	28.86	7.39	2.14	13.60	17.15	28.20		2.86
	37.25	3.21	1.12	2.30	30.37	25.48		0.27
Indiana <sup>b</sup> .....	23.92		8.00	12.34	21.48	Not given		
	28.08		8.64	12.18	19.90	Not given		
Slag: <sup>c</sup>								
Wisconsin .....	38.0	11.0	1.0	19.0	23.0		0.5-0.8	
Slag: <sup>c</sup> .....	38.4	10.5	0.7	15.3	31.5		1.6	
Rock wool: <sup>d</sup>								
Indiana .....	36.40		12.16	18.77	32.67			
Indiana .....	34.07		11.19	10.57	44.08			
Slag wool								
German <sup>e</sup> .....	34.0	9.1	2.7	6.2	38.7			2.3 <sup>f</sup>
American <sup>g</sup> .....	39.5		37.6	7.0	15.1			0.8
American <sup>h</sup> .....	36.3	13.1	20.4 <sup>i</sup>	2.4	27.2			0.6

<sup>a</sup> Fryling: ref. 11, pp. 161, 165 to 167.    <sup>c</sup> Guttman: ref. 16.

<sup>b</sup> Logan: ref. 22, p. 5.

<sup>f</sup> Fryling: ref. 11, p. 188.

<sup>c</sup> Thoenen: ref. 31, pp. 7 and 11.

<sup>g</sup> Includes MnO, 4.7 per cent; CaS, 3.8 per cent.

<sup>d</sup> Logan: ref. 22, p. 7.

<sup>i</sup> Probably exists in slag as FeO.

Natural woolrocks and rock mixtures, or composite woolrocks, are of two kinds, consolidated and unconsolidated. It is important that the former yield only a minimum amount of "fines" or small rock fragments in the process of quarrying and crushing, because fine materials tend to obstruct the free passage of air and heat through the cupola, thus reducing its efficiency, or are blown out by the air blast. The fact that unconsolidated materials produce fines is probably not important and they may even be desirable, as these materials generally require pre-cupola consolidation. This may be effected by various means, such as sintering, the use of cementitious or binding materials, or forming by pressure.

As the properties of rock wool are closely related to the chemical composition of the raw material from which it is made, it is desirable that woolrock deposits be of uniform character. Likewise, uniformity of sources of raw material for mixtures or composite woolrocks is important. Natural sedimentary woolrocks made up of a single type of rock, although variable in composition, are thought to be somewhat less variable as a



rule than those composed of interbedded, unlike rock strata, because the beds of the latter are often of variable thickness.

*Natural Woolrocks.*—Natural woolrocks may be divided into two general groups: (1) those comprising a single type of rock, and (2) those made up of a series of thin, interbedded, dissimilar rock strata that together, but not individually, have the proper  $\text{CO}_2$  content. Among the consolidated rocks in the first group are limestones and dolomites high in noncalcareous constituents, such as sand or silt grains, small irregular masses of silica or clay particles, more or less uniformly disseminated through the rock. Rocks of this kind may offer some advantages for making rock wool over rocks composed of larger lithologic units, as cherty limestone, because of the natural intimate mixture of their constituents.

Other consolidated rocks in the first group are certain types of conglomerates and cherty limestones or dolomites having a matrix comparatively high in carbonates and depending chiefly on the chert for the noncalcareous component. Or the matrix may be impure because of the presence of clay, quartz sand or disseminated silica, and thus contribute to the noncalcareous content of the material.

In the first group are also calcareous or dolomitic sandstone and shale. Sandstones of this nature usually are well cemented because the calcareous or dolomitic material present occurs at least in part as interstitial filling between sand grains and thus binds the grains together. Calcareous or dolomitic shales contain their carbonates either as relatively large masses such as fossils or nodules, or as small grains intimately mixed with the shale particles. Some shales of the latter type resemble impure limestones and dolomites in appearance and it is believed that in general they will occur in thicker, more solid strata than shales of the former type.

The unconsolidated rocks in the first group include the unindurated equivalents of the consolidated rocks mentioned above. Some glacial tills belong in this category, as do some calcareous or dolomitic clays, sands, gravels, marls, et cetera.

The second group of natural woolrocks—that is, woolrock consisting of interbedded strata of unlike lithologic character but of such composition that deposits of them have the chemical composition of woolrock, are commonly interbedded shale and limestone or dolomite, sandstone and limestone or dolomite or various other combinations of noncalcareous and calcareous rocks. Frequently, in such associations of rock types none of the types are “pure,” the shales being calcareous or sandy, the sandstones being clayey or calcareous and the limestones and dolomites sandy or clayey. The unconsolidated rocks in this group include the unindurated equivalents of the consolidated rocks.

Some igneous and metamorphic rocks, such as certain peridotites, pyroxenites, talcose and serpentinous rocks, contain the requisite amount

of lime and magnesia together with an approximately equal amount of silica and therefore are suitable from the standpoint of composition for making rock wool. However, their iron content is commonly high and special care would be required to ensure a white wool.

Certain minerals, such as wollastonite and some of the amphiboles, pyroxenes, talcs and serpentines, are of satisfactory chemical composition for making rock wool.

*Mixtures or Composite Woolrocks.*—Mixtures for making rock wool may be compounded of a large number of different earth materials, including sedimentary, igneous and metamorphic rocks. For example, dolomite and clay, limestone and quartzite, calcite and quartz, or bog lime, clay or sand, in the proper proportions should yield composite woolrocks. In brief, any calcareous rock, consolidated or unconsolidated, containing more than about 65 per cent calcium carbonate or calcium and magnesium carbonates, may be combined by suitable processes with consolidated or unconsolidated, siliceous or argillaceous rocks in such proportions as to give a composite woolrock.

*Deleterious Materials in Woolrocks and Slags.*—Of the many substances that may adversely affect the color, odor, or stability of slag or rock wool, the most common are compounds of iron and sulphur. These may be introduced as impurities occurring in the woolrock, slag or coke used in making the wool. While complete information is lacking regarding the deleterious effects of these substances, it has been demonstrated experimentally that the concomitant presence of iron and sulphur yields wool ranging from light tan through brown to black.

The presence in rock or slag wool of small amounts of calcium sulphide, which may result from the existence of gypsum in the raw materials, is also undesirable, as on hydrolysis this sulphide may generate bad odors and substances corrosive to steel. Small quantities of calcium sulphide are probably unavoidable in slag and rock wools, and it is impossible to state at present what is the permissible upper limit of concentration.

The substitution of lime by ferrous oxide in amounts up to 18 per cent produces low-temperature slag wools, which resemble in composition those produced from lead-furnace slag (see Table 2, last entry). These may be almost pure white. It is evident that the temperature of the molten material must be kept low, perhaps about 800° C., and that the reducing action of the hot gases in contact with the slag must be carefully controlled. In mineral wools produced at higher temperatures, a dirty gray color may result from the reduction of relatively small amounts of ferrous oxide to finely dispersed metallic iron.

The brown coloration attributed to iron sulphide may be prevented by proper operation of the cupola. It is instructive to compare this operation with that of a blast furnace. Important differences are: (1) the cupola is smaller; (2) its charge can be considered as a very low-

grade iron ore containing from 1 to approximately 3 per cent iron oxide; (3) its by-product is iron and not slag; and (4) it is operated to secure a high grade of molten slag (mineral wool), and not iron. When it is recalled that the blast-furnace manager can shift the sulphur between pig iron and slag at will, it becomes evident that by an intelligent application of the common principles of ferrous metallurgy a colorless mineral wool can be produced. Briefly summarized, it appears necessary: (1) to work under conditions such that the ferrous oxide is completely reduced to iron, (2) to work at temperatures sufficiently high for the small amount of dispersed iron so obtained to melt, coalesce, and separate from the slag, and (3) to work under such conditions that the reaction  $\text{FeS} + \text{CaO} + \text{C} = \text{CaS} + \text{Fe} + \text{CO}$  proceeds to the right. That the last of these conditions is important is suggested by one operator, who reports that when the wool begins to look brown he taps off the molten iron and thereby corrects the color.

*Distribution of Deposits.*—As rock wool can be made from any wool-rock that contains the requisite ingredients in proper proportions, or from artificial combinations yielding mixtures of suitable physical and chemical character, the variety of raw materials is too great for their distribution to be discussed here. So far as natural woolrocks are concerned, specific reports have been published regarding their occurrence in Indiana,<sup>22</sup> Illinois,<sup>20</sup> and Ontario, Canada.<sup>14</sup> Reports by various state geological surveys on limestone resources also furnish many data.

*Materials Used for Making Rock Wool.*—Only incomplete data are available regarding the many kinds of material employed in the United States. In the Indiana rock-wool district, in the central part of the state, an impure dolomitic limestone is used. An average of 20 analyses shows 31.37 per cent silica-alumina-iron oxide and 31.90 per cent lime and magnesia.<sup>22</sup> A more highly calcareous limestone is used as a flux to assist the melting of the woolrock.<sup>22</sup> In Illinois, rock wool is being made from dolomitic limestone and clayey siltstone and the production of rock wool from a deposit of limestone with which is interbedded relatively thin shale strata has been carried through tests employing a pilot plant. It is reported that at Netcong, N. J., silica rock and crystalline calcite are used (ref. 31, p. 9), and at Torrence, Calif., a mixture of limestone and silica refuse from ceramic plants with fluorspar as a flux.<sup>22</sup> Wollastonite from Kern County, California, melted in an electric-arc furnace has been shown experimentally to be a suitable material.<sup>22</sup> Tremolite,<sup>22</sup> hornblende, and soapstone<sup>24</sup> are also reported as materials for making rock wool.

#### PRODUCTION AND MARKETING

Recent information regarding the production of mineral wool and of limestone for use in making it is as follows:<sup>24</sup>

The Bureau of the Census reported figures on value of "mineral-wool products" manufactured in the United States based upon its canvass of asbestos-products manufacturers. In 1929, eight establishments—two in Indiana and one each in California, Illinois, Michigan, New Jersey, Ohio, and Wisconsin—reported a total output of rock- and slag-wool products valued at \$2,377,324. Production in 1931 was valued at \$2,873,230, and a preliminary figure of \$1,714,171 for 1933 was described as "incomplete."

The figures in the following table, although they indicate only the amount and value of the limestone used in making mineral wool, afford an index to recent growth, and show a strong upturn in 1928 and a well-sustained output after 1929, when demand for other building materials was at low ebb.

TABLE 3.—*Limestone Used for Rock Wool in the United States, 1919-33\**

Year	Short Tons	Value	Year	Short Tons	Value
1919...	12,500	\$17,164	1927...	15,390	\$12,962
1920...	21,570	24,773	1928...	31,700	27,042
1921...	b	b	1929...	83,920	92,092
1922...	9,170	10,622	1930...	64,850	70,938
1923...	6,940	9,333	1931...	73,640	67,393
1924...	7,530	14,908	1932...	43,180	38,321
1925...	12,660	14,308	1933...	55,160	42,305
1926...	18,880	19,448	1934...	68,020	54,087

\* From reports of limestone producers to the Bureau of Mines.

b Bureau of Mines not at liberty to publish figures.

According to the National Rock and Slag Wool Association: "During 1934, 26 companies in the industry reported a volume of \$3,695,-404.95; and during the year 1935, 30 companies reported a volume of \$5,571,468.62."

*Tests and Specifications.*—In general there are no satisfactory, commonly accepted tests or specifications for rating the quality of mineral wool, although the formulation of such standards is recognized as highly desirable by consumers and also by producers. Heat-conductivity measurements have been used as a basis for judging the quality of wool, but complaints are made that values obtained by different laboratories do not check. Consumers and producers of mineral wool should encourage research leading to the adoption of standard tests and specifications for: color, average fiber and shot diameters and the permissible variations from the average, length of fibers, percentage and characteristics of dust and flaky material present, percentage of shot, percentage of oil present, behavior of the wool at elevated temperatures, resistance to attack by acids and alkalis, amount of hydrogen sulphide generated on treatment with acids, and heat conductivity.

Typifying the specifications now extant, the following are those of one consumer for rock wool to be used at fairly high temperatures:

The rock from which the wool is produced shall contain no metal, sulphur or substances that conduct electricity or are corrosive to metals; the shot content shall not be in excess

of 15 per cent by weight, this measurement being obtained by shredding the wool and sifting through a 10-mesh sieve; and the wool shall contain no water, oil, or materials that give off offensive odors when heated.

*Fabrication, Uses and Marketing.*—Most of the mineral wool manufactured is used in forms that do not involve special fabrication. It has been found desirable usually to incorporate a small amount of oil with the wool during the manufacturing process, to reduce dust, make the product feel softer, and to a certain degree waterproof the material. However, oil-treated wool cannot be used for high-temperature insulation. Wool that is collected by an endless chain apparently has a natural tendency to pack together in the form of a bat, and will retain this form if treated with a binder such as sodium silicate. Granulated wool is made by treating ordinary wool in a modified threshing machine. The process removes all of the shot and coarse fibers and improves the appearance of the wool. Granulated wool brings a somewhat higher market price than ordinary wool because there is a loss of material in processing and therefore its cost is greater.

The industrial applications of mineral wool as a heat-insulating material are numerous. It is used for insulating pipes, boilers, tanks and walls, which serve as heat shields. It is also used in various manufactured products such as roofing, electrical refrigerators, automobiles, gas stoves and domestic appliances. One large outlet is afforded by manufacturers of refrigerating cars and the use of air conditioning in passenger trains provides a new outlet for the material. Mineral wool is used as a filter for acid liquids. It also finds extended use as a sound-insulating and sound-deadening material. For this, it may be used in a loose form or in the form of bricks for lining ceilings and walls.

The wide variety of uses to which mineral wool is put is responsible for the diverse forms in which it appears on the market. Granulated wool is blown between the walls for insulating houses. Loose wool and loosely formed bats are applied during the construction of buildings and also are used for wrapping pipes and tanks. By incorporating mineral wool with inorganic binding materials the various insulating plasters are produced. Wool can be sewed between paper or chicken wire to form blankets or held between expanded metal lath to form insulating boards. It is also incorporated in various manufactured products such as papers, boards, and felts, where its presence is not immediately apparent. Rock cork is fabricated in the form of blocks of considerable rigidity. Apparently this material is made by impregnating wool with an asphaltic emulsion. The details of such processes of treating mineral wool are generally trade secrets.

At the present time mineral wool in both loose and fabricated forms is handled by building-supply dealers, lumber yards, roofing concerns, etc. There are companies that specialize in applying granulated wool

to houses already constructed. Some of these dealers require wool in bulk form; others require it in such form that the quantities handled can be designated in terms of board-feet.

*Prices.*—Published information on the price history of mineral wool is meager. The industry was summarized in *Mineral Industry* for 1911 for a 12-year period, during which time the value per ton ranged from \$8.40 to \$11.28. It is believed that these figures refer mostly to slag wool. In 1929, Thoenen gave the price of raw rock wool as from \$20 to \$25 per ton, granulated wool up to \$40 per ton. Blankets were listed as from 8 to 10¢ per board-foot and blocks at 15¢ per board-foot. Undoubtedly the prices given for raw wool and granulated wool were low. While the Rock and Slag Wool Code was in effect a uniform retail price of about \$50 per ton was maintained throughout the United States for mineral wool in bulk and the price of the granulated material refined for blowing into houses was approximately \$70. It is understood that the tendency of prices since the termination of the Code has been slightly downward. In 1923, Saborsky gave 12¢ per pound as the cost of glass silk produced by spinning. The price of glass wool produced by the newly developed blowing method is considerably lower than that.

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## CHAPTER XXI

### ICELAND SPAR AND OTHER CRYSTALS OF RELATED USE

BY H. HERBERT HUGHES,\* MEMBER A.I.M.E.

**I**CELAND spar is a variety of calcite (calcium carbonate) characterized by its purity, transparency, and perfection of crystalline structure. Although actual demand is small, it has no substitute in polarizing microscopes and other scientific instruments.

#### PROPERTIES AND USES

Iceland spar is relatively soft; its hardness is 3 in Mohs' scale. The specific gravity is 2.713. To be suitable for optical uses each crystal or cleavage fragment must be at least one inch long and one-half inch thick. Each piece must be colorless and absolutely transparent, completely free from cloudy inclusions, cavities, or foreign particles. The material must not show any internal iridescence due to incipient cracks along cleavage planes. Even twinning lamellae invisible to the naked eye may ruin some specimens for optical use. Imperfections or impurities may not be scattered throughout the entire crystal, however, and in some instances it may be possible to take advantage of the perfect rhombohedral cleavage to trim away imperfect portions and recover cleavage fragments of satisfactory spar.

Twinning of Iceland spar crystals is common. It usually can be detected by moving the crystal slowly back and forth over a dot on a sheet of paper. If the crystal is normal two dots will appear and vary little in their relative positions upon movement of the crystal, but if the crystal is twinned, additional indistinct dots may also appear in various positions.

The strong double refraction of Iceland spar is utilized in the nicol prism, a device that transforms an ordinary light ray into plane-polarized light. A cleavage rhombohedron of Iceland spar about three times as long as it is broad is cut diagonally along the plane connecting the obtuse dihedral angles of two opposite elongated faces, and the end surfaces are ground down until they are perpendicular to this newly formed plane. The two halves are then cemented together with Canada balsam, a transparent cement having an index of refraction lying between

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\* Mineral Economist, U. S. Bureau of Mines, Washington, D. C.



the values of the indexes of the two rays common to calcite. The cemented nicol is polished and then set in cork, blackened along the contact with the calcite in order to absorb light reflected to the sides. Immediately upon entering the nicol, any ray of white light is broken into two rays, the ordinary and the extraordinary. The ordinary ray has a higher index of refraction, therefore it is refracted to a greater extent than the extraordinary and meets the Canada balsam at an angle so great that it is reflected from it and is absorbed by the blackened walls of the prism. The extraordinary ray, because of its lower index of refraction in this direction through the crystal, meets the Canada balsam at a smaller angle and passes through it without being appreciably affected. This ray emerges from the opposite end of the nicol as plane-polarized light with its vibration plane parallel to the short diagonal of the rhombic section of the prism, and polarized in the plane parallel to the film of Canada balsam. This vibration plane is rigidly fixed by the crystalline structure of the calcite. Modifications of this style of nicol have been devised, but they all embody the principle of totally reflecting either the ordinary or the extraordinary ray.

By means of the petrographic microscope, which uses two nicol prisms, the behavior of polarized light in traversing thin sections of minerals can be studied. The optical properties thus revealed provide an accurate means of identification for most minerals. Iceland spar, therefore, is of inestimable value in the scientific investigation of rocks and minerals. Nicol prisms or modifications of them are used in other scientific equipment that aids manufacturers in keeping a close check on the quality and uniformity of their products. Among these instruments are saccharimeters, used to measure the sugar content of a solution; colorimeters, which indicate depth of color as compared with a standard; photometers, for measuring intensity of light; dichroscopes, which reveal dichroism in crystals; polariscopes, and polarimeters, which are used in the study of polarized light; spectrometers, used in determining index of refraction; and spectroscopes, for forming and examining spectra. Iceland spar is used also in the determination of crystal structure by X-ray analysis.

Only a small percentage of the spar recovered from most deposits is of optical grade, and unfortunately there is practically no market for material that is too cloudy or otherwise too imperfect for optical use. Many fine crystals of Iceland spar have been acquired by various museums, but this market is in direct competition with optical manufacturers. Numerous smaller specimens are sold to museums, colleges, schools, or private collectors, either as additions to mineral collections or for demonstration of optical properties.

A small quantity of chemically pure Iceland spar is consumed by manufacturing chemists in the preparation of standardizing solutions for use in volumetric analysis.

## ORIGIN AND DISTRIBUTION

Iceland spar is formed by deposition of calcium carbonate from water solution, but the conditions necessary for the formation of transparent spar rather than ordinary calcite are not definitely known. No blanket statement covering geologic occurrence of the material can be made. In general, however, the principal deposits are associated with basic igneous rocks, occurring as masses or crystal aggregates in cavities of the country rock. These pockets are usually filled with residual clay. The belief that the calcite has been deposited from magmatic rather than meteoric solutions has many supporters, but there is little evidence to support the theory. Other minor deposits of Iceland spar, usually of inferior quality, occur as vein material in limestones and related rocks.

Deposits of Iceland spar have been reported in many of the Western States, and small quantities have been produced near Greycliff, Mont., Cedarville, Calif., and possibly at other operations. Except for a period during the World War, however, when supplies of spar as well as manufactured prisms from Germany were cut off, manufacturers have not depended upon domestic deposits.

The spar deposit in Iceland, not far from Eskifjordur, was discovered during the early part of the seventeenth century, but little attention was given it earlier than 1650, and actual development of the property was not begun until 1850. At that time part of the deposit was privately owned and part was owned by the Government, but in 1879 the Government obtained entire control, and all operations since then have been under governmental supervision, either directly or by lease to native or foreign companies. In 1919 the pit was 100 ft. long and 72 ft. wide at the top. The average depth was 52 ft., and the bottom dimensions were about 55 by 50 ft. No definite order or system of development was followed, the clay pockets being worked out by hand with picks and shovels wherever they were found. The largest clay pockets containing the most perfect crystals were found in the southwestern corner of the pit. Undercutting in this corner left a dangerous overhang of rock, which made further progress in that direction extremely hazardous, and it was recommended that the open-pit method of mining be abandoned in favor of underground development.

The spar crystals from the Iceland mine are variable in size, and, as in every deposit, many are too imperfect for optical use. In addition to cracks and distortions, the principal imperfections are air bubbles, included water, and streaks of clay or other foreign mineral matter. Operations were discontinued for several years during the World War, and the quarry was flooded to protect the crystals from weathering. When it was reopened, the quality of the material was below the standard of the prewar product, therefore the deposit has been worked only inter-

mittently, and consumers have been forced to seek elsewhere for supplies of spar for optical use.

Recent demand for Iceland spar has been supplied principally from South Africa. The most important locality has been the Kenhardt district, northwest Cape Province, where numerous deposits are scattered over a large area. The spar occurs as irregular masses of various sizes embedded in soft, sandy clay, which fills cavities in weathered diabase. The spar is recovered solely by hand methods. Iceland spar probably was discovered in South Africa first by early explorers, but its value was not recognized until about 1920, when small shipments were sent to Germany. Most of the material is sold to German manufacturers, but it has also been imported into the United States during the past few years. Production apparently is not continuous, but various persons have been working the material intermittently since 1922 or earlier. Spain also has supplied spar of good quality within the past few years, and deposits in other countries have been reported. According to present indications, however, South Africa may be expected to supply the bulk of the demand for spar of optical grade.

#### MINING METHODS, CONSUMPTION AND PRICES

*Mining Methods.*—Ordinary open-pit methods of mining have been used in most Iceland spar operations. The country rock is broken by explosives, after which the clay pockets are worked out by hand to recover the spar crystals. These, in turn, may be broken by small hand hammers into pieces that can be handled more readily. Iceland spar is easily shattered, and this rough treatment undoubtedly has been responsible for a large proportion of the incipient fractures in the finished product. The use of explosives in mining this material should be avoided, for even though the masses of spar may be partly cushioned by the clay surrounding them many of the crystals are ruined by blasting. Regardless of the mining method selected, the fragile nature of the material demands extreme care.

An interesting departure from conventional quarrying methods was reported to have been used in California, involving the use of unslaked lime rather than explosives in breaking the rock surrounding the crystals. Three or four inches of unslaked lime is tamped in the bottom of a hole drilled a short distance from the crystals. A slender iron rod or wire is inserted into the hole, and lime is packed around it to within 2 in. of the top, by means of a curved tamping bar. The rod is then removed, and a cotton string saturated with water is dropped into the hole so that the end is suspended about  $\frac{1}{2}$  in. off the bottom. The opening of the hole is then sealed with clay or some similar material. The water dripping from the string causes expansion of the lime, which bursts the rock with a minimum of damage to the spar.

*Consumption and Prices.*—Before the World War, European companies, particularly in Germany, supplied virtually all the nicol prisms and other Iceland spar parts used by American manufacturers of scientific apparatus. During the war, however, rapid depletion of stocks of finished prisms forced the American companies into the manufacture of these essential parts. The present domestic production of nicol prisms is being supplemented by imports, and there has been a ready market, both in the United States and abroad, for high-grade optical spar.

No production or consumption figures for Iceland spar are available, but the quantity actually sold in the United States is small, probably not more than 200 or 300 lb. a year. The volume of foreign consumption is considerably greater, no doubt.

No definite price for Iceland spar can be quoted. In many transactions a fair price is arrived at by negotiation between the buyer and seller. The principal factors influencing price are the quality of the spar, the size of the fragments or crystals, and the quantity to be sold. The status of the reserve supplies of the purchasing company exerts a pronounced influence on prices; a manufacturer with a large reserve of optical spar will not pay as high a price as another manufacturer whose supplies are nearly exhausted. The price of spar of optical grade during the past several years has fluctuated from \$7 to \$35 a pound. Both of these limits are extreme—\$10 to \$25 probably would be more nearly normal. Seasonal trends and general business depressions have little effect on Iceland spar.

The average price for museum or collection specimens is about \$1 to \$3 a pound, although the value of extra fine crystals is equivalent to that of optical-grade material. Standardizing spar usually sells for \$1 to \$2 a pound.

#### OTHER MINERALS IN OPTICAL APPARATUS

*Tourmaline.*—A crystal of tourmaline also transforms a ray of light into any ordinary ray vibrating perpendicular to the principal axis and an extraordinary ray vibrating parallel to this axis. Plates cut parallel to the principal axis, when placed at right angles, cut off all light. This simple polarizing device, known as tourmaline tongs, is rarely used except to demonstrate polarized light.

*Fluorite.*—Fluorite (calcium fluoride) is used extensively in metallurgical, chemical, and ceramic industries. The mineral has a low index of refraction, disperses light faintly, and normal crystals display no double refraction. This unique combination of properties accounts for its use in lenses and lens systems to correct spherical and chromatic errors. Fluorite of optical grade is used in spectrograph prisms and other optical apparatus where transparency of the ultraviolet and infrared parts of the spectrum is essential. It has also been used in lens systems of

telescopes to correct undesirable color effects. Water-clear fluorite is preferred for optical use, but faintly tinted crystals may be suitable. Common flaws due to incipient cleavage, bubbles or other inclusions render the material unsatisfactory. Every commercial fluorspar operation is a potential source of optical-grade material. Prices vary widely, depending upon the size of the flawless crystals or cleavage pieces.

*Quartz*.—A quartz wedge, cut from a crystal of quartz usually in such a manner that one flat side is parallel to a plane containing the optic axis and the long edge inclined at an angle of  $45^\circ$  to the optic axis, is an important accessory to a polarizing microscope. This device is used to determine the slow ray and the optical sign of thin sections of minerals. Flawless, transparent crystals of quartz are required for the manufacture of quartz wedges but their use is limited.

*Selenite*.—Another accessory used with the polarizing microscope is the sensitive plate. This device, made from a clear cleavage piece of selenite (gypsum), will reveal even the slightest double refraction in mineral sections.

*Mica*.—A cleavage piece of mica may be used with the polarizing microscope to determine the optical sign of a mineral section.

Use of these common minerals in such optical devices are of scientific interest but obviously of little importance to the quartz, gypsum, or mica industries.

*Artificial Compounds*.—Polaroid, a synthetic material recently developed, acts in the same manner as Iceland spar in polarizing light rays, but apparently it has not been used in optical apparatus. Suggested uses include an interesting application in the automobile industry. A section placed in each windshield in the line of vision of the driver oriented at right angles to similar sections in approaching headlamps would eliminate all glare from night driving. The tremendous task of supplying every existing automobile with this equipment precludes all possibility of early adoption of the material, but its use would certainly be a forward step in safeguarding highways.

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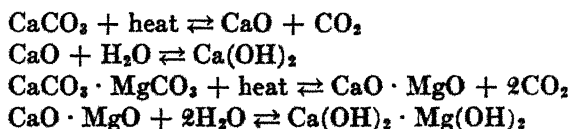
## CHAPTER XXII

### LIME

BY PAUL HATMAKER,\* MEMBER A.I.M.E.

**L**IME is a substance resulting from the calcination of limestone, dolomite, or other calcareous material. As commonly used, the term properly includes both quicklime and hydrated lime. Quicklime is composed chiefly of calcium oxide (CaO) or calcium oxide in natural association with a smaller amount of magnesium oxide (MgO), capable of slaking with water. Hydrated lime is a dry powder obtained by treating quicklime with enough water to satisfy its chemical affinity under the conditions of its hydration. It consists chiefly of calcium hydroxide  $[\text{Ca}(\text{OH})_2]$  or a mixture of calcium hydroxide and magnesium oxide and magnesium hydroxide.<sup>2</sup>

Commercial limes may contain, as impurities, as much as one or two per cent silica, several tenths of one per cent of oxides of iron and aluminum, and sometimes traces of sulphur, phosphorus, sodium, potassium and rarer elements. Carbon dioxide, identified with uncalcined raw material, may also be present in small quantities. With calcite and dolomite as raw materials, the manufacture of lime is exemplified by the following equations:



These equations assume pure materials and ignore reactions of lime during the burning process, with impurities always present in commercial stone, which sometimes affect properties of the finished product.

#### PROPERTIES

The utility of lime is dependent upon both its physical and its chemical properties, which in turn are closely related to the properties of the stone or other calcareous material from which it is made. The properties of

\* National Lime Association, Washington, D. C.

The author wishes to express his appreciation of the many helpful comments and suggestions by Messrs. W. V. Brumbaugh, Lee S. Trainor and H. A. Huschke, of the staff of the National Lime Association.

<sup>2</sup>References are at the end of the chapter.

lime, furthermore, are influenced by time and temperature of calcination, and those of hydrated lime by conditions of slaking.

Quicklime may be white to light gray or light buff in color, when properly burned from limestone of a high degree of purity. It is earthy in appearance, apparently amorphous (but possibly cryptocrystalline) in character, and is porous. The specific gravity is  $\pm 3.3$  and varies only slightly among different limes. The apparent density (grams per cubic centimeter), however, is more variable, ranging from 1.15 to 2.09 for a number of representative samples of lump quicklime tested in the laboratory of the National Lime Association. The average pore space is  $\pm 55$  per cent, 55.4 per cent being an average of 74 samples tested. Quicklime has great affinity for water, but there are wide variations in rates of slaking of different limes. High-calcium quicklime generally slakes more rapidly, with greater evolution of heat, than does a high-magnesium lime.

Hydrated lime is a very fine white powder, with a specific gravity of  $\pm 2.3$  and an apparent density of  $\pm 0.85$ . The average particle size is about 6 microns in diameter.

Calcium oxide is refractory, with a melting point of  $\pm 2570^{\circ}$  C.

Some of the major uses of lime are dependent upon properties difficult of precise measurement. In agriculture, for example, lime (either in the form of oxide or hydrate) not only acts as a neutralizing agent upon the soil acids, but it also has the property of flocculating or "loosening" clayey soils, thus increasing the tilth.

In construction, lime is valuable because of its physical properties. Lime putty—hydrated lime with an excess of water to form a pasty mass—is highly plastic, which is one of its chief virtues in mortars and plasters. It also possesses cementing properties due possibly to hydroxyl crystal formation, but chiefly through recarbonation of the hydroxide and a reversion to the carbonate form. Lime mortars also are evaluated on the basis of sand-carrying capacity, putty yield from the quicklime, water-retaining capacity, and "workability," the latter no doubt being dependent upon and perhaps synonymous with water retention and plasticity. These properties of mortar are closely related to the characteristics of the lime itself.

In the chemical industries, lime is an inexpensive and widely used base and causticizing agent, and is an important reagent in a host of industrial processes. As amplification of the properties of lime that make it of such wide commercial use cannot easily be divorced from description of the uses themselves, a fuller discussion will be given under the section Marketing and Uses. Both properties and ultimate uses of lime are related to manufacturing processes and the kind of limestone burned, and thus may be affected by factors of origin and occurrence of the raw material itself.

## ORIGIN AND MODE OF OCCURRENCE

Limestone is the major source of calcium carbonate from which commercial lime is derived. In fact, most lime in the United States is made from either limestone or dolomite, although some production is recorded from oyster shells and deposits of calcite. Many limestones are highly fossiliferous, some appearing to be composed chiefly, if not entirely, of the remains of marine organisms. Many different varieties of sea life, such as foraminifera, mollusks and corals, secrete calcium carbonate to form skeletal structures, which ultimately accumulate on the sea bottom. Eventually there are thus formed great deposits of calcareous matter composed largely of skeletal remains consolidated and cemented into compact, relatively homogeneous rock formations.

Erosion of land masses and the formation of sediments in the sea are concomitant with and perhaps form one cause of diastrophic forces that raise new continents from what were ocean bottoms. Geologic history is replete with these continuing cycles of erosion, deposition, emergence and submergence, and many calcareous deposits of the sea bottoms have become limestone formations of land areas.

Calcium carbonate is only slightly soluble in pure water, but solution is relatively rapid in water containing carbon dioxide. Part of the load of drainage waters emptying into the sea is calcium carbonate that has been dissolved from rocks containing calcium salts over which the waters have traveled. Upon reaching the ocean, some of the calcium carbonate may be precipitated because of its lesser solubility in sea water or by ammonium carbonate resulting from decaying organic life. The lime salts remaining in solution may be used in the skeleton-building processes of marine life. Deposits such as calcareous tufa and travertine are formed by direct chemical precipitation of the dissolved calcium carbonate from water.

The origin of dolomite is obscure, but most authorities believe it is formed chiefly by direct chemical action of waters high in magnesium and calcium salts. One reason for this theory is that skeletal remains of only a few sea animals contain appreciable quantities of magnesium. Dolomite deposits may be fossiliferous, for subsequent replacement of calcium carbonate by magnesium salts may preserve the original fossil structure.

Limestone deposits present almost infinite degrees of consolidation, from marl or chalk, in which the calcareous skeletons are but loosely cemented, through the many types and kinds of compact limestone to marble, where through heat and pressure the calcium carbonate has become dense and highly crystalline with no physical trace of the original fossil constituents.



Purity of the limestone is of greater importance to the lime manufacturer than the physical aspects of the stone or than the structural mode of occurrence. Only very small amounts of silica, alumina, or other impurities can be tolerated, inasmuch as the proportions of them virtually double when the stone is calcined. So-called "siliceous," "cherty," "argillaceous," or "ferruginous" limestones, containing considerable quantities of silica, clay or shale, and iron, as a rule are not suitable for the manufacture of the usual commercial grades of lime.

The character of the overburden offers important operating problems, for in open quarry workings the overlying clay, soil or other material provides two sources of contamination of the limestone. The first source of pollution is through the medium of bedding planes, jointing planes, solution channels, or other openings whereby ground waters carrying silica, alumina and iron salts held either in solution or in suspension penetrate through the limestone, depositing such material in the channels traversed as well as forming encrustations on the rock surfaces. The second source of foreign material during actual quarry operations is clay, silt, or other dirt that may wash into the quarry from the surface, carrying small quantities of iron, silica or alumina to the stone. Impurities also may occur in the limestone itself, frequently as small grains of silica, nodules of chert, minute pyrite crystals, or other distinct mineral constituents. Without considering the nature of impurities that may be introduced during subsequent manufacturing operations, there are these three sources of compounds other than lime, which must be recognized and dealt with during quarry operations. It is apparent that contamination through weathering agencies becomes of less import when mining methods are used; although solution channels may occur in limestone formations many hundreds of feet below the top of the water table.

The limestone deposits of this country contain infinite variations in chemical analysis; their age geologically is from the pre-Cambrian to the Quaternary or Recent, with all the variations in fossil content that this implies; their physical properties are of endless variety; their structure varies from unaltered and undisturbed sedimentary beds to deposits faulted, folded, jointed, and inclined at any angle; their form may be anything from marl or oyster shells to marble; and subsequent weathering may have been inconsequential or it may have formed sinkholes, solution channels, and caves wholly or partly filled with secondary depositions of foreign material. A particular deposit of stone is worthy of intensive geologic study if the raw material of limemaking is to be well understood, and if such knowledge is to be translated finally into better finished products.

The physical nature of the stone itself is important both from the standpoint of manufacturing processes and properties of the finished product. Hard, dense, compact stone, for example, may calcine more

quickly or at lower temperatures than more porous material. Some types may crumble during burning; others may become "overburned" easily, and require particular vigilance throughout the burning process.

Calcium carbonate, in any form, yields calcium oxide and carbon dioxide when heated to the point of calcination. Furthermore, calcium oxide, from whatever source, will combine with water, or slake, forming, with increased amounts of water, first lime putty and then milk of lime. Here similarities seem to stop. Rates of slaking vary, and properties of quicklimes as well as of lime putty and milk of lime differ greatly, depending upon the manufacturing processes and the chemical and physical properties of raw limestone itself.

The varying properties of different limes undoubtedly are influenced by such differing characteristics of the limestones as kind, form, quantity of impurities, fossil content, density, porosity, and degree of crystallization. Perhaps traces of metallic elements or compounds, some of which commonly are disregarded in ordinary methods of analysis, have an unsuspected effect upon the properties of lime products. Abundant opportunity for study and research exists in the field of the behavior of lime products and its relation to the different properties of limestone, as well as the effect thereon of manufacturing technique.

#### DISTRIBUTION OF DEPOSITS

Limestone occurs in every state of the United States, and deposits have been used for the commercial manufacture of lime in all but six states. Many regions, however, are of comparatively minor importance because either the deposits are relatively unsuitable for lime burning or markets are remote. Cost of fuel also may be a factor in some places. In 1935, according to the Bureau of Mines, 18 states produced more than 92 per cent of the lime shipped, and eight states alone accounted for nearly three-fourths of the production for that year. A number of the more important producing localities may be mentioned briefly in the order of their present relative volume of sales.

*Ohio.*—Thick, flat-lying beds of dolomite are found close to the level surface of northwestern Ohio, slightly south and east of Toledo. This district produces most of the hydrated lime used in the United States in the finishing coat of plaster, these deposits producing a very white, highly plastic hydrate, which has enjoyed nation-wide distribution. Important plants are situated near Genoa and Clay Center in Ottawa County; Gibsonburg and Woodville in Sandusky County; at Luckey, Wood County, and Carey, Wyandot County. This district produces a wide variety of lime products, and several plants manufacturing chiefly dead-burned dolomite for refractory purposes are in this general vicinity. Calcium limestone occurs in Ottawa County and is quarried and burned

at Marblehead. Other important deposits of calcium limestone occur at Columbus and near Delaware, and also at Pedro, near Ironton, in the southern part of the state. Magnesium lime also is produced near Springfield.

*Pennsylvania.*—At one time, more than 800 plants produced lime from the widely occurring deposits of limestone in this state. The industry, however, is now chiefly centered at or in the general vicinity of Bellefonte, York, Annville and Cedar Hollow. High-calcium lime is produced from the Bellefonte area from steeply inclined beds. The York and Annville districts also produce high-calcium products; whereas the Cedar Hollow deposits are dolomitic. A deposit of dolomite near York is used for the manufacture of dead-burned dolomite for refractory purposes.

*Missouri.*—High-calcium lime is produced from three important localities in Missouri—near Hannibal, near Springfield, and in the vicinity of St. Louis, at Ste. Genevieve. Deposits are flat-lying, easily accessible by open quarry or mining methods, and the products are distributed widely, particularly among the industrial markets.

*West Virginia.*—The principal lime-producing region of West Virginia is in the Martinsburg area, in the extreme northeastern portion of the state. Both high-calcium and high-magnesium lime products are manufactured.

*Tennessee.*—A number of lime plants are situated near Knoxville, and considerable high-calcium waste material from the marble quarries is used for manufacture of lime. A high-calcium limestone deposit at Sherwood is also an important source of production.

*Virginia.*—The Shenandoah Valley has been an important lime-producing region for many years. Production has centered around Strasburg and Riverton at the north, Eagle Rock in the central part and Ripplemead at the southern end of the valley. Production is chiefly high-calcium material, although deposits of high-magnesium stone also are utilized.

*Alabama.*—The Alabama lime industry is chiefly in the Birmingham district, where extensive deposits occur of both high-calcium and high-magnesium limestone.

Other major lime-producing points (in the order of the 1935 production by states) have been developed in the vicinity of Chicago, Quincy and Alton, in Illinois; at Mitchell, Ind.; near Farnams, Mass.; at Buffalo, N. Y.; near San Francisco and Los Angeles, Calif.; near Milwaukee, Wis.; in the Frederick area of Maryland; at Dallas, Houston, New Braunfels, and El Paso, Texas; near Rutland, Vt.; near Petoskey and Menominee, Mich.; and on San Juan Island, in Puget Sound.

Lime is also produced in Maine, Connecticut, Rhode Island, New Jersey, North and South Carolina, Georgia, Florida, Kentucky, Minnesota, Iowa, Oklahoma, Arkansas, Louisiana, South Dakota, Colorado, New Mexico, Utah, Arizona, Nevada, Montana, Idaho, and Oregon,

but all these states together produced less than 8 per cent of the 1935 production.

#### POLITICAL CONTROL AND FOREIGN TRADE

Limestone is abundantly available over the surface of the earth, deposits suitable for limemaking occurring in many countries. Furthermore, lime is a bulky and relatively low-priced commodity, and is perishable when in the form of quicklime. These factors minimize extensive shipments and tend to make lime a minor element in world trade. Lime is an essential raw material for many industries, whether in times of peace or war. The United States, however, has virtually unlimited reserves of limestone suitable for making high-grade commercial lime. Manufacturing facilities in 1935 are estimated to have been nearly three times the market demand for that year.

Imports and exports of lime are insignificant from a national viewpoint, exports amounting to 3710 tons, and imports 17,268 tons in 1933, according to data published by the Bureau of Mines. Imports come chiefly from Canada to the State of Washington, where, however, such foreign competition is of relatively great importance to domestic producers in that region. Under the terms of the Trade Agreement between Canada and the United States effective Jan. 1, 1936, the duties on lime are as follows:

Imports from Canada: Lime not specifically provided for . . .	7c per 100 lb.
Hydrated lime . . . . .	8c per 100 lb.
Exports to Canada: Lime not specifically provided for . . .	22.5 per cent ad valorem.
Hydrated lime . . . . .	11c per 100 lb.

Exports from the United States go to many countries, the Dominican Republic, Peru, Canada, Japan, Panama, Mexico and the Philippine Islands being among the thirty or more countries of destination. The total quantity exported is relatively small, amounting to only a few thousand tons annually.

#### PRODUCTION, CONSUMPTION, AND STOCKS

The largest volume of production ever recorded by the Bureau of Mines was 4,580,823 short tons in 1925, with a total value f.o.b. plant, exclusive of package costs, of \$42,609,141. Conversely, the smallest volume was 1,959,990 short tons sold in 1932, with a total value of \$12,302,231. Sales have increased steadily since this low point of the depression, and the volume in 1935 is estimated to have been 2,952,000 short tons valued at \$21,428,000. Particularly in recent years, the total capacity of the industry has tended to keep well ahead of actual production. Table 1, with total capacity figures as computed by the National Recovery Administration from data furnished by the Bureau of Mines, is illuminating in this regard.

TABLE 1.—*Shipments of Lime and Capacity of Plants<sup>a</sup>*

Year	Total Shipments, Short Tons	Total Capacity, Short Tons	Ratio, Shipments to Capacity, Per Cent
1917	3,786,364	7,158,000	52 9
1921	2,532,153	8,284,000	30 6
1925	4,580,823	8,318,000	55 1
1929	4,269,768	9,424,000	45 3
1933	2,269,230	8,232,000	27 6

<sup>a</sup> Data include dead-burned dolomite used as a refractory. The total capacity figures are computed on the basis of 365 days a year, continuous kiln operation.

Lime is sold for a wide variety of uses. Table 2 shows the relative importance of the several markets in 1934.

TABLE 2.—*Lime Sold by Producers in the United States in 1934, by Uses<sup>a</sup>*

Use	Quantity <sup>b</sup>		Value	
	Per Cent of Total	Short Tons	Total	Per Ton
Agricultural . . . . .	9 3	222,077	\$1,478,128	\$6.66
Building. . . . .	21.3	511,419	4,260,865	8.33
Chemical:				
Glassworks . . . . .	3 2	76,232	507,031	6.65
Metallurgy . . . . .	14.0	334,406	2,003,788	5.99
Paper mills . . . . .	12 3	293,798	1,899,053	6.46
Refractory lime (dead-burned dolomite) . . . . .	13.5	324,868	2,698,414	8.31
Sugar refineries . . . . .	0 9	20,528	183,098	8.92
Tanneries . . . . .	2.6	62,745	484,410	7.72
Water purification . . . . .	7.6	183,581	1,229,435	6.70
Other uses <sup>c</sup> . . . . .	15 3	367,433	2,419,802	6.59
Total chemical . . . . .	69 4	1,663,591	11,425,031	6.87
Hydrated lime (included in above totals) . . . . .	100.0	2,397,087	17,164,024	7.16
	34.6	829,430	6,324,623	7.63

<sup>a</sup> Data from U. S. Bureau of Mines.

<sup>b</sup> The total quantity includes lime used by producers (captive tonnage) to the extent of 129,290 short tons valued at \$671,864.

<sup>c</sup> Includes a multitude of chemical and industrial uses.

The lime industry is fortunate in many respects in serving such a diversified market. Both durable and nondurable goods are represented among the various uses. Building construction and steel (the latter accounting for much of the metallurgical and refractory lime) are outstanding among the durable-goods industries, and it has been well said that they are either prince or pauper. In boom years, such industries

are highly active, and in times of depression they are relatively dormant. The quantity of lime sold to these industries fluctuates accordingly, varying with great sensitivity with steel output and volume of construction.

On the other hand, many industries using lime produce consumer or nondurable goods. People eat, drink water, and wear out shoes even at times when they do not build homes. Certain lime markets, such as water-purification plants, sugar refineries, tanneries and agriculture, are less subject to cycles of booms and depressions. These conditions afford large potential markets for lime in periods of prosperity as well as certain "backlogs" in times of depression.

The character of the market demand also affects seasonal production. Much of the agricultural lime is sold during a few weeks in the spring, and a lesser peak occurs late in the summer. Building lime usually is in greatest demand during the spring and summer months. Lime plants, therefore, generally are most active from early spring to late fall.

Stocks of lime products are never a major factor in the industry. Quicklime being perishable, in that it quickly absorbs moisture and carbon dioxide from the air, is not stocked by the manufacturer to any extent. Hydrated lime may be stored for reasonable periods under favorable conditions before recarbonation becomes serious. Limited stocks may be accrued to help level off seasonal peaks of demand, but beyond this operators prefer to shut down their plants rather than to accumulate large stocks of finished products. Hydrated lime in bulk or in bags housed in a tight, dry warehouse may be stored for a year without appreciable damage. Specimens of regular hydrated lime in ordinary commercial paper packages have been under observation at Virginia Polytechnical Institute for over 10 years, during which the rate of recarbonation has been carefully checked and recorded. During the first year or two, the reaction with carbon dioxide proceeds in the outer inch or so of the mass, after which the rate of recarbonation is definitely slowed up. Specimens under observation for 10 years show the outer two or three inches partly recarbonated but the interior of the mass not greatly affected.

#### PROSPECTING AND EXPLORATION

The areal geology of the country has been well developed by the United States Geological Survey and the various state institutions charged with surveying and mapping geological formations. Much general information, therefore, is available in regard to the position, extent and composition of limestone deposits. A given limestone formation, however, may have locally a dolomitic, ferruginous, cherty, or other variable phase of its more common characteristics. Intensive localized geological study may be repaid by the discovery of an important commercial deposit where none was hitherto suspected; such study, moreover, is essential in determining adequate reserves prior to capital expenditures, a proper

plan of exploitation, and in maintaining reserves sufficiently in advance of actual production—points sometimes overlooked with embarrassing, if not disastrous, results.

The usual methods of geological field work are useful in reconnaissance surveys. Limestone outcrops, highway and railroad cuts, caves, sink-holes, well logs, and soil and subsoil content may reveal much information not only as to structure but as to chemical and physical characteristics. Surface stripping, pits, or trenches may be employed to determine in more detail the conditions at and near the surface, and where used should be augmented by rock excavations to points below action of weathering agencies.

Drilling frequently is done to determine reserves beyond the quantity actually exposed or "in sight." Churn drills afford a relatively inexpensive and generally available method of securing samples of the deposit. Spacing of the holes will vary with local conditions, but they should always be sufficient in number to eliminate any risk of substantial changes in formation between holes. Core drilling may be used where it is desired to obtain greater knowledge as to the physical character of the deposit, or where the beds dip so steeply as to necessitate inclined test holes.

Proper development of a limestone deposit requires the same careful attention to the nature and extent of reserves as would be given to a deposit of a metallic ore. It is perhaps needless to say that such exploration work should have competent technical supervision, for otherwise it might be quite ineffectual, and subject to incorrect interpretation.

In appraising the economic possibilities of a limestone deposit, it should be remembered that a lime-manufacturing operation is a logical source of rock products such as stone aggregates, railroad ballast and pulverized limestone. Furthermore, impure limestone that could not be used for making lime may offer possibilities when such products are considered as mineral wool, masonry cement, and natural or Portland cement. Some manufacturers have been highly successful in developing various products, using limestone as a raw material, and are achieving not only a diversified output but marked operating economies and minimum waste as well.

#### MINING AND QUARRYING METHODS

Limestone deposits occur under a great many different structural conditions, each of which requires local adaptation of extraction methods. In former years, open quarries were the rule, but now many lime companies have changed to underground mining methods because stripping became so excessive or overhanging walls became so unsafe that the usual quarry practice was impractical.

Quarry methods involve stripping the overburden, drilling the exposed rock, blasting, loading and transportation of the broken stone. Stripping

may be done with steam shovels, teams and scrapers, or by hand methods. The method adopted will depend upon the quantity of material to be moved, the degree of consolidation of the overlying dirt, and to some extent upon the topography of the underlying rock, which may be eroded by sinks, pot holes and the like, which require much hand labor. Sometimes a layer of rock must be stripped in order to expose the desired formation, and drilling and blasting must be done. Stripping may be done in two stages. In the first, the bulk of the overburden may be removed well ahead of quarrying; and in the second stage, the rock surface is cleaned off perhaps a relatively short distance ahead of the quarry face.

A number of working faces are necessary to provide flexibility to the operation. With horizontal beds, the desired result is accomplished by advancing a thin bed over a long face, or a thick bed in benches over a shorter face, the dimensional terms here used being, of course, relative. With vertical or steeply inclined beds, less stripping is necessary in proportion to potential tonnage of stone exposed, but it is more difficult quickly to provide "elbow room," and sometimes it takes years properly to develop an open quarry following a vertical or steeply inclined formation with somewhat variable conditions of composition and structure.

Churn drills may be used for large blasts, but in lime quarries most drilling is done with air drills, either mounted or not. Blasting is so regulated as to minimize fines. Pop-holing or mud-capping is employed to break up large boulders after blasting, and much hand sledging is generally necessary to achieve a maximum quantity of one-man stone for the kilns.

Unless the stone is very uniform, considerable hand selection is necessary while loading. Fines as well as waste stone may thus be rejected or used for purposes other than limemaking. In large, highly mechanized quarries, steam shovels and railroad cars and locomotives are used. Loading is generally by hand in the smaller quarries, and tramming may be by hand or other means. Tractors and specially designed dump-car trailers provide a highly flexible method of handling stone where conditions permit. This system obviates the necessity and expense and the bother of track work. In some quarries, however, inclined tramways are necessary because of extreme differences in elevation between the quarry floor and the plant.

The general quarry plan may have as its prime objective the production of stone uniform in quality, or it may be desirable to produce different grades of stone for making different grades of lime. The various factors relating to problems of quarrying limestone for lime manufacture have been discussed in detail by Bowles and Myers.\*

Room-and-pillar methods may be used in underground mining of flat-lying deposits. Such mines generally are continuations of open quarry operations, and require a minimum of developmental work. A



modified shrinkage-stopping system is used at one mine, where the deposit of limestone is steeply inclined. Such a plan requires the usual development through shafts, drifts, raises, et cetera.

Both mining and quarrying methods have inherent advantages, depending upon local conditions. Thoenen has summarized the various factors influencing a decision as to open quarry versus underground mining (ref. 37, pp. 22 and 28):

The advantages of mining may be summarized as follows:

1. Removal of large masses of overburden is avoided.
2. A mine may be operated the year round, irrespective of weather conditions, thus affording steady employment to workers and promoting contentment in the personnel and a low labor turnover.
3. Under certain conditions storage facilities can be provided underground more easily than on the surface and without deterioration of the product. The surplus produced when the demand is slack can be stored in anticipation of peak demands.
4. The clean stone produced at mines often commands a higher price than quarried stone. In fact, cleaning, at considerable expense, is often necessary to make the quarried product salable. Moreover, the limestone strata in mines are not exposed to the action of frost or water or mud washed from a clay overburden.
5. For fluxing, mined stone is cleaner both in lump and fine sizes than quarried stone.
6. A mine can be advanced to property lines regardless of such factors as extent of overburden, dip of strata, cavities, and surface conditions. Moreover, mines can be worked within city limits, where quarrying might be dangerous or be forbidden by ordinances.

On the other hand, there are certain disadvantages, which also must be considered.

1. Some means of ventilation must be supplied to carry off fumes from explosives and furnish fresh air to workers.
2. There is danger from falling rock.
3. More fines are produced than in a quarry.
4. The type of labor required is more skilled, and therefore more expensive, than quarry labor.
5. Costs are in general higher than for quarries, but the difference may be counterbalanced by the higher quality of the product.

#### PREPARATION FOR MARKET

The manufacture of lime products includes the selection and sizing of the raw limestone, the burning or calcination of the stone to drive off the carbon dioxide, inspection of the kiln product, with removal of any core (uncalcined material), and overburned lime, size gradation, and loading into cars where sold as bulk quicklime. Quicklime, however, may be hydrated, or pulverized, and packaged, stored for a limited period, or shipped immediately.

Certain markets require limes of specific properties. A given quarry may contain stone of varying characteristics, so that different properties in the finished product may be obtained by proper selection of stone in the quarry. Some deposits, however, are so uniform in composition that any variations in character of finished product must be achieved through special burning conditions.

Stone selected for the kilns must be sized. Vertical kilns commonly use material from about fist size to chunks as large as a man can lift easily—called “one-man stone.” Material of this nature is commonly produced by blasting and sledging in the quarry. For rotary kilns, the stone is crushed and graded with minimum size about  $\frac{1}{2}$  in., and the maximum generally not more than  $1\frac{1}{2}$  or 2 in.

Lime kilns vary in size and design. The idea of rotary kilns was borrowed from the cement industry, but the modern vertical kilns have developed from the first primitive “heap-burning” of stone where wood and stone were piled together. Enclosing walls of crude stone masonry marked the next step. Pot kilns with a mixed feed of fuel and stone evolved into the “patent” or shaft kiln, where the stone is charged at the top and lime is drawn at the bottom, the fireboxes being apart from the shaft itself.

Vertical kilns are designed to provide several distinct steps in the burning process. The first is the preheating of the kiln feed, whereby the cold stone charged into the top of the kiln is gradually brought up to the calcination temperature by contact with the hot gases from the burning zone. After the stone is heated to about the calcination temperature (about 898° C. for calcium carbonate and 756° C. for dolomite) it is kept within the burning zone long enough for most or all the carbon dioxide to be expelled. This is the critical point in lime manufacture. Properties of the finished product may be greatly affected by both the temperature of the burning zone and the length of time that the stone is held at the maximum temperature.

After passing the firebox level, the fragments of hot quicklime begin to cool, although there is somewhat of a “soaking” action. The lower part of the kiln acts as a cooler, so that the quicklime, drawn at intervals usually of 4 to 6 hr., can be inspected, forked, and perhaps loaded immediately into boxcars for bulk shipment.

Wood is an ideal fuel because of its relatively long, cool flame, but inadequacy of supply, or comparatively high cost, has led to extensive use of coal, either alone or supplemented by wood. A coal fire may be modified by such other means as introducing steam in the firebox, or recirculating part of the stack gases through the burning zone. The object in each case is to control combustion, to distribute heat more evenly and effectively throughout the mass of stone fragments, and to avoid points or zones of excessive temperature. Other fuels include natural gas, fuel oil and producer gas.

The rotary kiln is simply a refractory-lined cylinder which may be 60 to 200 ft. long mounted on trunnions to permit rotation. It pitches slightly toward the discharge end. A shorter revolving cylinder serves as a cooler.

Fuel efficiencies of kilns vary widely, depending upon composition of the rock, design of kiln, kind of fuel used, and the skill of the kiln

A considerable number of data on factors relating to rates of settling of lime suspensions have been developed by Holmes, Fink, and Mathers.<sup>14</sup> From many tests on different kinds of lime, the following relationships were indicated:

1. Suspensions of magnesium limes as a class settle much more rapidly than do suspensions of calcium limes.
2. Those limes, both calcium and magnesium, that as a class have a high available lime content, settle most slowly. Exceptions were noted, however, to this conclusion.
3. Among the calcium limes, the freshly slaked materials settle more slowly than suspensions made from mechanically hydrated limes. (Other observers have pointed out that excess water with calcium quicklime produces slow settling and highly reactive suspensions, and as much as 10 times the theoretical amount of water necessary to hydrate the material has been recommended in one case for best results.)
4. Lower temperatures of slaking tend to produce slower settling rates. After-heating, if not excessive, tends to increase the settling rate.
5. The effect of increased time of soaking of the mechanically hydrated material is to decrease the rate of settling.

#### SAMPLING AND TESTING LIME PRODUCTS

Methods for sampling and testing lime products have been adopted by the American Society for Testing Materials, and full details will be found in the volumes published every third year by the Society.

#### SAMPLING

Methods of sampling are covered by A.S.T.M. Designation C-50, and conform generally to usual sampling technique. Triplicate samples are taken either at point of manufacture or at destination. Bulk material must be sampled from at least 10 different parts of the mass, the total quantity so taken to be at least 100 lb. for each 30 tons of material, and never less than 50 lb. This must be crushed, if necessary, to pass a 1-in. ring. Selection may be by shovels with lump or granular material, or by a sampling tube with minimum diameter of 1 in. for powdered material. When in packages, at least 2 per cent of the number of packages (never less than five) are sampled. With lump or granular material, the containers selected are emptied and the contents passed through a 1-in. ring. With powdered material, at least 1.5 per cent of the packages (never less than five) are sampled by a tube with minimum diameter of  $\frac{1}{2}$  in., taking material the length of the container. Such selections are followed by thorough mixing and quartering to provide three final samples of at least 5 lb. each for every 30-ton unit, and never less than 2 lb. These samples must be sealed immediately in airtight, moisture-proof containers.

## CHEMICAL ANALYSIS

Standard methods of chemical analysis of limestone, quicklime and hydrated lime are described in detail under A.S.T.M. designations C-25 and C-110. Usual analyses include determination of mechanical moisture, total volatile matter or loss upon ignition, silica and other insoluble matter, iron, alumina, calcium oxide, magnesium oxide, carbon dioxide, sulphur, phosphorus, strontium and manganese.

"Available lime" is a common term used in evaluating the chemical properties of lime products for certain purposes. The term refers to the constituent or constituents entering into a chemical reaction under the conditions of the specified method or process. Available lime is commonly determined by the modified Scaife method, whereby the reactivity of a sample with hydrochloric acid is measured by use of phenolphthalein as an indicator, and the results expressed in terms of "available CaO." Available lime is sometimes determined by the sugar method, whereby a small sample of material is treated with a solution of pure, neutral cane sugar in water free from carbon dioxide and the amount of lime thus dissolved by the sugar solution is determined by neutralizing with a standard acid using phenolphthalein as an indicator.

Still another method of judging the value of a given lime for certain purposes is to determine its chemical reactivity with sodium carbonate expressed in terms of percentage of causticization. The sample of lime is added to a solution of sodium carbonate. At desired intervals portions are pipetted off, filtered, and titrated with standard acid to the end point with phenolphthalein, and then to the end point with methyl orange to obtain the total alkali.

## PHYSICAL TESTS

Physical characteristics of lime about which information is commonly desired include rate of slaking, percentage of residue, fineness of hydrate, settling rate, and such characteristics of lime putty for construction purposes as soundness and plasticity. Tests for these properties are largely empirical, and may at times be supplemented according to the problem at hand. Commonly used methods will be described briefly; most of them either are recognized or have been adopted as standard by the A.S.T.M. in connection with standards of the Society for lime for structural purposes.

*Rate and Methods of Slaking.*—As an appendix to the standard specification for quicklime for structural purposes (A.S.T.M. designation C-5) the Society makes certain recommendations in regard to the preparation of lime putty. To determine the rate of slaking of different limes, it is suggested that several lumps of quicklime the size of one's fist be placed in a bucket and enough water be added to just cover the lime. If slaking

begins in less than 5 min., the lime is "quick slaking"; from 5 to 30 min., "medium slaking"; over 30 min., "slow slaking."

Quick-slaking limes are not easily drowned. To slake, the lime should be added to the water and great care should be exercised to prevent burning. Spots emitting steam should be hoed, and if necessary more water should be added. Medium-slaking limes may be slaked in a similar way, but care must be taken not to add too much water. With slow-slaking limes, water is added to the lime, which is moistened thoroughly. After the reaction has started, water may be added a little at a time, but not so that the mass is cooled. Hoeing is deferred until the reaction is practically complete.

For "white coat," the putty is passed through a No. 10 sieve and stored for a minimum of two weeks. For base coats, the putty is run through a No. 8 sieve. Sand up to equal parts by weight, and all the hair required, are added, and it is stored for a minimum of two weeks. For mason's mortar, all or part of the sand is added, and it is stored for a minimum of 24 hours.

*Percentage of Residue.*—Quicklime for structural purposes (A.S.T.M. designation C-5) is tested for the percentage of residue by carefully slaking a 5-lb. sample and washing it for not more than 30 min. through a No. 20 sieve. The residue is dried to constant weight at a temperature of 212° to 225° F.

*Fineness of Hydrate.*—Hydrated lime for structural purposes (A.S.T.M. designation C-6) is tested for fineness by placing a 100-gram sample on a No. 30 sieve nested above a No. 200 sieve and washing the sample through with water for no longer than 30 min. Residues on each sieve are dried to constant weight in an atmosphere free of carbon dioxide, at a temperature between 100° and 120° C. The weight of the residue on the No. 30 sieve is combined with that on the No. 200 to obtain the correct weight of material retained on the No. 200 sieve.

*Rate of Settling.*—The method used in the laboratory of the National Lime Association to determine the rate of settling of lime suspensions was to slake carefully 7½ grams of quicklime to a putty of normal consistency. After a 24-hr. curing period, the putty was transferred to a 100-c.c. graduate with an internal diameter of 23 mm., and water was added to the 75-c.c. mark; the graduate was well shaken, and more water was added to bring the volume to 100 c.c. when the graduate was again shaken for 30 sec. Standing undisturbed, the volume of the lime suspension as it settled was read at 5-min. intervals until the 50-c.c. mark. Final volume after 24 hr. was also read. With hydrated lime, a 10-gram sample was mixed with water to form a putty, which was allowed to soak for 24 hr. This was then transferred to the graduate and the same procedure followed as with quicklime.

*Soundness.*—Under standard specifications for structural hydrated lime (A.S.T.M. designation C-6) a method is described for testing the constancy of volume or soundness of a lime. Such a test is designed primarily to guard against popping or pitting of plaster due to incomplete hydration of the oxides at time of use. In making the test, a sample of the lime is mixed with standard Ottawa sand to make a good plastic mortar of rather dry consistency. From this a pat is made and placed in a closet for 24 hr. at a temperature of between 65° and 75° F. It is then soaked in water until a film stands unabsorbed on the top. A thick cream prepared from a separate sample is spread upon the surface and the whole dried again as previously noted. It is then suspended in steam at atmospheric pressure for 5 hr. and allowed to cool for at least 12 hr. If the steam has no visible effect upon the pat, the material is considered "sound." The time involved in making this test lessens its usefulness from the viewpoint of manufacturing control, but no satisfactory accelerated test has as yet been developed.

*Plasticity.*—The plasticity of a lime is commonly determined in the United States by use of an Emley plasticimeter. This apparatus consists essentially of a porous porcelain base plate so mounted as to hold a truncated cylinder-shaped mass of putty against a movable disk with an indicator to measure torque applied thereto. The base plate absorbs water from the putty, and the objective is to measure the varying torques translated from the base plate through the putty to the disk. The base plate is rotated very slowly (one revolution in 6 min. and 40 sec.) and has an upward travel of  $\frac{1}{16}$  in. per revolution. The plasticity figure is calculated by formula from the torque developed upon the upper disk, and the time in minutes until the end of the test, or until the sample is no longer a coherent mass. Details of this plasticity test are contained in the Standard Specifications for Hydrated Lime for Structural Purposes (A.S.T.M. designation C-6).

A device has been developed in England by which a cone of putty upon the platform of a standard flow table is subjected to a series of knocks until it has spread to a certain diameter; the greater the plasticity, the more knocks are necessary. Further details may be obtained from publications of the Building Research Station, Department of Scientific and Industrial Research, Watford, England.

#### MARKETING AND USES

Lime is widely used in agriculture, construction, and industry. Because these three broad market divisions differ greatly not only in the way in which lime is utilized, but in many of the ramifications of marketing and distribution, they will be discussed individually

## AGRICULTURE

Lime is applied to the soil principally to correct soil acidity and to improve tilth. It acts as a soil conditioner.

Soils are composed essentially of the residue from the weathering of rock formations commingled with accumulated organic matter resulting from the decomposition of vegetable and animal life. Although calcium is one of the most abundant elements in the earth's crust, most virgin soils, except in arid regions, are acidic. This condition is caused by the normal processes of weathering and leaching. Streams and rivers constantly carry away enormous quantities of lime in solution. The cycle of moisture movement from the clouds to the earth to the oceans to the clouds is a process of constant removal of calcium salts from the topsoil. Even in a limestone country, the soil itself may be deficient in lime because of this leaching process. The effect of leaching, however, is offset to some extent by lime salts in rain water, which may bring as much as 100 or 200 lb. of calcium carbonate or its equivalent to an acre of land in a year. This increment in lime content from rain might exceed the loss through leaching in cases of very deficient soils, but losses of lime from leaching generally are considered to be several times the gain from rain. Natural conditions, therefore, tend toward the creation and maintenance of strongly or moderately acid soils.

Where topsoil is undisturbed, the resulting plant growth inhibits erosion, reduces leaching, and slows up the rate of loss of calcium salts. Owing to the possible presence of residual lime carbonates and the increment from dust, rainfall, and decayed vegetation, the rate of acidification may be substantially retarded, if not entirely stopped. Cultivation, moreover, accelerates leaching and erosion, and causes losses of lime removed with the crops. Few soils in regions of moderate to heavy rainfall (such as prevail in the eastern half of the United States) can long withstand this increased withdrawal of lime without replenishment from other than natural sources. Since most of the arable land in the eastern part of this country has been under cultivation for 75 to 200 years or more, it is not surprising that liming has become a necessary part of the technique of soil management.

The chief functions of lime on the soil are commonly recognized as being: (1) to reduce soil acidity, (2) to supply calcium and magnesium as plant foods, (3) to granulate the soil, rendering it more tillable, (4) to promote decay of organic matter and the formation of nitrates, (5) to increase the efficiency of fertilizers, and (6) to counteract certain soil poisons.

Liming materials include lime, raw limestone, and sometimes other calcareous materials. A common measure of the effectiveness of a liming material is the available oxide content. Particle size, however, is

also important. The following table shows the approximate range of available oxide content of a number of liming materials:

MATERIAL	AVAILABLE LIME OXIDES PER TON, LB.
Lump quicklime.....	1400 to 1900
Ground or pulverized quicklime ..	1400 to 1900
Hydrated lime.....	1200 to 1600
Pulverized limestone, shells, and marl ..	800 to 1100
Limestone screenings ..	400 to 700
By-product materials ..	350 to 600

A vast amount of experimental work has developed facts as to the optimum degree of soil acidity for different crops. Most crops grow best where the soil reaction is not more than slightly or moderately acid. A few, such as alfalfa, sugar beets and sweet clover, definitely favor a neutral or only slightly acid soil. Very few crops prefer a strongly acid soil. Simple soil-testing equipment enables anyone quickly to determine the acidity of a soil, and the pH value thus derived can be translated easily in terms of pounds of lime oxides necessary to make the proper soil correction. Abundant data are available from various agricultural experiment stations pertaining to all phases of liming.

Lime is generally distributed to farmers through dealers in agricultural commodities. It may also be sold direct, or through farm cooperative organizations. In lime-producing localities, farmers may buy lime at the plant. Some lime companies carry on intensive selling and promotional campaigns, including such services as soil testing, the dealer merely carrying the individual farmer accounts and maintaining warehouse facilities.

According to data compiled annually by the National Lime Association, the quantity of lime oxides used in 1935 per acre of cropped land varied from 65.0 lb. in Wisconsin and 62.0 lb. in New Jersey, to 0.3 lb. in Kansas, with 15 states either without data as to consumption or using no liming materials.

### CONSTRUCTION

The principal uses of lime in construction are in masonry mortars, in stucco, and in base and finishing coats of plaster. Lime is also used as an admixture in concrete and in whitewash or other cold-water paints.

*Masonry Mortar.*—The essentials of good masonry construction are a sound foundation, carefully selected masonry units, and a mortar that under usual field conditions will bind the units into a solid, watertight, monolithic construction. Laboratory research in the field of mortar materials during the past few years has thrown considerable light upon



the factors affecting masonry construction. Many data have been gathered relating to volume changes of mortar resulting from repeated wetting and drying and changes in temperature, absorptive qualities of masonry units, water-retaining capacities of various mortar materials, strengths of bond between the mortar and the unit, and usual data as to strength of masonry under compression, tension and flexure. Results of much laboratory work show why, for thousands of years, lime has been an ideal mortar material, since a high lime content ensures a workable, plastic mortar of high water-retaining capacity and excellent bonding properties—a mortar readily adaptable to different kinds of masonry units and ensuring watertight walls.

Volume proportions commonly recommended are two parts hydrated lime or lime putty, one part Portland cement, and not more than nine parts of sand for above-grade masonry construction; below grade, a one lime, one cement, and six sand mix is recommended.

*Plaster.*—Lime has been used as a plaster material for almost as long as there are records as to building materials. It is used in scratch and brown-coat work as well as in the finishing coat. The highly competitive construction field offers markets for a host of substitute materials for interior wall finishes, but lime has the advantage of greater economy in material cost and makes a plaster that is more easily troweled, has desirable acoustical properties, is noncorrosive to metal, and is well adapted to any standard backing or subsequent decoration. Its chief disadvantage has been its relatively slower time of setting, which has diminished the use of lime plaster on construction work where time was an important factor.

Lime plaster consists of a properly aged lime putty to which is added sand and such hair or fiber as may be required. Plasticity of the putty is an important prerequisite, particularly for the finish coat. Furthermore, the lime putty must make sound plaster, free from subsequent pitting or popping caused by inadequate aging of the putty or improperly prepared lime.

*Stucco.*—Lime is widely used in exterior plaster work over metal lath on frame, brick, tile, or other materials. Lime putty to which has been added sand (and hair for the scratch coat) may be applied in three coats, much as in interior plaster work. Portland cement may be added to give greater initial strength and more rapid rate of hardening. Usual proportions of the mix are one part lime putty and three parts sand. For scratch-coat work, 1 bushel of hair is added to 1 cu. yd. of sand. When a cement is added, it may displace the lime up to an amount not exceeding one-fourth of the volume of the lime.

*Concrete.*—Lime is used to some extent as an admixture to concrete, the usual proportions recommended being from 5 to 8 lb. of lime for each

sack of cement. A more workable, denser, and slightly stronger concrete is said to be made possible by thus adding lime. With greater density and less segregation of aggregates, there results less patching and pointing up of the job and less eventual penetration of water.

*Whitewash.*—Many formulas have been devised for making whitewash, of which the following is an example of one simple to prepare and of wide application: Soak 5 lb. of casein in 2 gal. of hot water for about 2 hr., or until thoroughly softened. Dissolve 3 lb. of trisodium phosphate in about 1 gal. of water, and add this solution to the casein. Allow this mixture to dissolve. Prepare a thick cream by mixing 50 lb. of hydrated lime in about 7 gal. of water, stirring vigorously. Dissolve 3 pints of formaldehyde in about 3 gal. of water. When the lime paste and the casein solution are both thoroughly cool, slowly add the casein solution to the lime, stirring constantly. Just before using, slowly add the formaldehyde to the batch, stirring constantly and vigorously; if added too rapidly the casein may jell, thus spoiling the batch. Only as much should be prepared as can be used in one day. Borax may be substituted for the trisodium phosphate if the latter is not available. For preparing a dry mix, zinc sulphate may be substituted for the formaldehyde in the proportion of  $\frac{1}{2}$  lb. to every 50 lb. of hydrated lime.

*Mortar Colors.*—A mixture of lime putty and sand generally is white, but some grades may be a very light cream or buff. Any pigments added should be insoluble in water and inactive with the lime. Mineral pigments commonly used are chrome oxides for greens, precipitated hydrated iron oxides for yellows, brown iron oxides for browns, ferric oxides for reds, ultramarine and cobalt blues for blues, cobalt violet for violets, and black oxide of iron for black.

*Constants.*—For estimating quantities of lime and lime putty, the following relationships will be found accurate enough for most practical purposes: For 1 cu. ft. putty, 27 lb. quicklime, or 44 lb. hydrate, or 94 lb. Portland cement.

Nine cubic feet of putty and 27 cu. ft. of sand are required for 1 cu. yd. of lime mortar, plaster or stucco.

*Specifications.*—The American Society for Testing Materials has developed certain minimum requirements for lime for structural purposes, covering both quicklime and hydrated lime. Quicklime shall conform to the requirements listed in Table 3 as to chemical composition, calculated to the nonvolatile basis. The residue on a No. 20 sieve, after slaking and washing through the screen, shall be not greater than 15 per cent by weight.

Specifications for hydrated lime cover both masons' and finishing lime, masons' lime including material used for scratch or brown coat of plaster, for stucco, for mortar, and for addition to concrete. For both

TABLE 3.—*Requirements for Quicklime*  
CALCULATED TO NONVOLATILE BASIS

	Calcium Lime	Magnesium Lime
Calcium oxide, minimum, per cent. ....	75	
Magnesium oxide, minimum, per cent. . . . .		20
Calcium and magnesium oxides, minimum, per cent.	95	95
Silica, alumina and oxide of iron, maximum, per cent. .	5	5
Carbon dioxide, maximum, per cent:		
Sample taken at kiln. . . . .	3	3
Sample taken elsewhere . . . . .	10	10

classes, the calcium and magnesium oxides must constitute not less than 95 per cent of the nonvolatile portion. Carbon dioxide may not exceed 5 per cent at place of manufacture or 7 per cent at any other place. The residue on a No. 30 sieve must be not more than 0.5 per cent and on a No. 200 sieve not more than 15 per cent. The lime must test "sound" when made into pats and steamed in accordance with the soundness test. Finishing hydrated lime must have a plasticity index of 200 or more.

*Markets.*—Sales of lime for construction purposes are directly affected by the peaks and depressions in general building activity. The largest markets lie in regions of greatest population density, although different communities, of course, will provide greater or lesser markets depending upon the quantity of building being done. Some variation in demand also will be noted among different communities, depending upon local building practices.

Chief substitute or competing materials for lime in the construction field are: Portland cement and various prepared masonry cements for mortar; and gypsum plasters and wallboards for interior walls.

Building lime is generally distributed through builders' supply dealers, although a substantial proportion is sold to jobbers by the manufacturer. Only a minor percentage is sold direct to the consumer or contractor. A comparatively recent development in the industry is the establishment of plants where quicklime in bulk is manufactured into putty, which is properly aged and sold, either alone or mixed with sand, directly to contractors.

#### INDUSTRIAL AND CHEMICAL PROCESSES

Lime is widely used in many industries as a flux, a refractory, or a chemical reagent. Owing to its low cost and general availability, it is pre-eminent as a base and causticizer. About two-thirds of the total

lime production of the country, amounting to about 2 million tons in 1935, was sold for chemical or industrial purposes at an average mill value per ton of \$6.92. The principal markets, with the percentage of total sales that they represented in 1934, are as follows: Metallurgy (chiefly open-hearth steel furnaces), 14.0 per cent; refractory (dead-burned dolomite), 13.5 per cent; paper mills, 12.3 per cent; water purification, 7.6 per cent; glassworks, 3.2 per cent; tanneries, 2.6 per cent; and "other uses," 16.2 per cent. Important in this miscellaneous group are alkali works, calcium carbide, coke and gas manufacture and insecticides, although a great many others provide substantial tonnage.

The number and the diversity of the functional capacities of lime give it a rich resourcefulness and potentiality for new and larger uses which it shares with no other material. No detailed description of the part played by lime in the industrial and chemical field can be given in this chapter, but the more important, salient features will be outlined by major use.

*Metallurgy.*—Metallurgical uses include fluxing material, used chiefly in open-hearth and electric steel furnaces, as well as lime used to condition the pulp in flotation-mill circuits and to aid in the cyanide process of gold recovery. The basic open-hearth process of steel manufacture is of major importance in the United States, owing to the characteristics of the iron ore used, which generally contains too much phosphorus and sulphur for refining by the Bessemer process. A 100-ton "trade heat" of the pig-iron scrap process requires about 55 to 60 tons of steel scrap, 55 to 60 tons of pig iron, 8 to 10 tons of limestone or lime, and 5 to 6 tons of iron ore. These proportions, of course, vary considerably, some plants using higher percentages of scrap metal and others having a continuous process whereby molten pig iron ("hot metal") is transferred directly from the blast furnaces to the open hearths. The impurities present, such as silicon, manganese, phosphorus and sulphur, pass into the slag, which should be of an oxidizing nature and strongly basic, containing an ample excess of free lime in order to retain the oxidized impurities. This excess of lime is generally kept somewhat under 55 per cent of the weight of slag. During recent years, the ratio of scrap to pig iron has been increasing from year to year, and there is a tendency to use a greater percentage of lime under these conditions. High-calcium lime is used as an open-hearth flux, and should contain as little silica and sulphur as possible, although no rigid specifications are in general use. The tolerance of impurities is predicated to some extent upon the cost of raw material and, therefore, purity of the lime used becomes in some degree an economic function of the price.

Lime has become an important flotation reagent, particularly since about 1923, when a change became apparent from the oil flotation with

acid circuits to chemical flotation with alkaline circuits and selected concentration of the minerals. Nearly all flotation is carried on in slightly alkaline pulps, as these are best for the chemical collecting agents.\* Large operating economies have been possible with this transition to alkaline solutions, inasmuch as the acid pulps were destructive of all iron with which they came into contact. A high-calcium lime is generally used in the flotation circuits and is added either as quicklime, hydrated lime, or milk of lime, choice being governed by local factors. Here again, precise specifications are largely a matter of the kind of material available and the requirements of the particular mill in question.

In the cyanide process, lime is used as a neutralizing agent to counteract the effect of soluble acid salts in the mill or mine water, as a coagulating agent to effect the settlement of slimes, and as a protection against the soluble and insoluble cyanicides in the material treated and any atmospheric carbonic acid that may be present.†

*Refractory (Dead-burned Dolomite).*—Dead-burned dolomite is used extensively as a refractory in basic open-hearth steel furnaces, basic Bessemer converters, as well as other high-temperature processes. Various processes have been developed for the manufacture of dead-burned material, but the essential principle is to calcine dolomite or a high-magnesium limestone to such a high temperature that a sintering action results, this being accomplished in some cases by a double burning of the material. Such material as iron oxide, alumina or silica sometimes is added to the dolomite better to increase the sintering action and to produce a material impervious to moisture. The dead-burned material is used both as an aggregate in preparing a monolithic refractory masonry lining and as a prepared brick shape, using tar or some other binding agent, which is fired and subsequently used as a masonry unit.

*Paper Mills.*—One of the most important methods of making paper from wood is the sulphite process, used mainly for the wood of coniferous trees, such as pine, spruce and hemlock. By digestion in an acid liquor under high temperature and pressure, all constituents of the wood chips except cellulose are dissolved and removed. The acid liquor is a solution of magnesium and calcium bisulphites, together with more or less free sulphur dioxide. It is obtained by treatment of either milk of lime or wet limestone, with sulphur dioxide prepared by burning sulphur or iron pyrites in air.

Dolomitic, or high-magnesium, lime, is preferred in the sulphite

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\* A. F. Taggart: *Handbook of Ore Dressing*, 845. New York, 1927. John Wiley and Sons.

† J. E. Clennell: *The Cyanide Handbook*, 537. New York, 1915. McGraw-Hill Book Co.

process, inasmuch as magnesium bisulphite is more stable, more soluble, and more effective in its chemical reaction than calcium bisulphite. The ratio of magnesium oxide to calcium oxide should be reasonably constant, and the lime moderately free from impurities. The chemical composition, calculated to a nonvolatile basis, should be a minimum of 94 per cent of  $\text{CaO}$  plus  $\text{MgO}$ , not more than 3 per cent combined oxides of iron, aluminum and silicon, and not more than 5 to 10 per cent of  $\text{CO}_2$ .

Lime is also used in both the soda and sulphate processes of paper manufacture and in the recovery of caustic soda, as well as in certain allied industries such as the manufacture of strawboard. Lime used in the soda and sulphate processes should be low in magnesia but both high-calcium and high-magnesium limes are used in the manufacture of strawboard.

*Water Purification.*—Lime is used widely in water-treatment plants, not only by municipalities but by industrial consumers such as manufacturing plants, railroads, and ore-treating mills. The usual objectionable impurities in water supplies for industrial or domestic use are mechanically carried inorganic particles, dissolved carbonates and sulphates of calcium and magnesium, and bacteria from various sources. Because of carbon dioxide absorbed from the atmosphere, ground water dissolves calcium and magnesium carbonates from the soil and rocks, producing what is known as "temporary hardness." When lime is added to such water, it combines chemically with the excess carbon dioxide, forming calcium carbonate, which, together with the calcium carbonate previously held in solution as bicarbonate, is precipitated and subsequently removed by filtration or decantation. As magnesium carbonate is only slightly soluble in water, additional lime must be added to convert it to magnesium hydroxide, in which form it is precipitated along with the calcium carbonate produced by the reaction.

Lime and soda ash are required to remove "permanent hardness" from water, which is caused by the presence of the sulphates of calcium and magnesium. Lime is also an effective coagulating agent, which assists in the settlement of material mechanically carried by the water in suspension. Lime possesses definite germicidal properties, and by proper use will render water safe from the standpoint of bacteria.

Either quicklime or hydrated lime of the high-calcium type may be used for the treatment of water. The American Water Works Association recommends in its specifications that quicklime should contain at least 85 per cent available  $\text{CaO}$ , and hydrated lime 90 per cent available  $\text{Ca}(\text{OH})_2$ .

*Glassworks.*—Lime is widely used in the manufacture of glass, functioning as the alkaline earth element in the batch. Silica in the form of sand is combined with an alkali-metal compound, usually sodium

carbonate or sodium sulphate, and an alkaline-earth compound, usually quicklime. Various other materials may be present, depending upon the kind of glass to be made. Either quicklime or hydrated lime of either calcium or magnesium type may be used, depending upon the method of production. Because of the close control of glass batches, it is highly important that the material furnished be constant in chemical composition. Even slight variations are objectionable, a tolerance not exceeding 2 per cent being sometimes stipulated in the purchase contract.

The chemical requirements of lime vary as to the kind of glass produced. For optical glass, the iron oxide should be practically zero whereas in bottle glass as much as 0.5 per cent is permissible, with nearly the same limits for blown or sheet glass. The silica or alumina may run as high as 15 per cent for bottle glass, but should be very much less for the other grades. Sulphur and phosphorus must be low in the anhydride forms, not exceeding 1 per cent for bottle glass and as low as about 0.2 per cent for optical glass. The combined CaO and MgO should be at least 89 per cent for bottle glass, 91 per cent for sheet glass, 93 per cent for blown glass, 96 per cent for rolled glass, and 99 per cent for optical glass. The quantity of carbon dioxide present should not be more than 3 per cent with quicklime and 5 per cent with hydrated lime. Quicklime generally is used in the pulverized form, and while specific sizes vary among different plants, the general limit is that it shall pass a 12-mesh sieve.

*Tanneries.*—Lime is used extensively in the tanning industry at the depilatory stage of leather processing. It not only is a desirable dehairing agent, but retards putrefaction and is not injurious to the hair removed from the hides. Ordinarily, either quicklime or hydrated lime with an available calcium hydroxide content of 85 per cent or better may be used. Quicklime should yield a fine, smooth putty, the suspensions of which should be extremely slow in settling at the milk of lime stage. High-calcium lime is generally used, but in making Morocco leather a high-magnesium lime may be preferred.

*Other Uses.*—The foregoing chemical and industrial uses of lime, while of major importance from the standpoint of tonnage requirements, represent but a very small number of the ways in which lime is used in the modern industrial world. There is virtually no chemical process wherein lime is not used in some stage, either directly or in the manufacture of the reagents themselves. Space prevents the fuller discussion or even mention of most of these uses, all of which are of more or less importance to the lime industry. Among those that it is believed will be of increasing importance in the future may be mentioned particularly the field of sewage disposal by means of chemical precipitation of solids.

## MARKETING AND PRICES

Chemical lime generally is sold direct to the consumer, sales through dealers being of relatively minor importance. True jobbers or brokers are somewhat of a factor, particularly in handling sales of the smaller producers, but probably more chemical lime is sold through agents or commission men than through those that take title of the shipment for purposes of resale. The big tonnage of this branch of the industry, however, is sold directly by the producer to the ultimate consumer, with no intermediate step in either negotiations or actual distribution.

Cultivation of the industrial lime market calls for a high degree of technical sales ability, for the producer may sell to many different customers in as many different industries, each having varying problems of utilization that must be well understood if the lime product is to render a full measure of service. Lack of knowledge of the properties of lime, as well as ignorance of the duty lime was supposed to perform, without doubt have retarded the wider use of lime in the chemical and industrial field.

Competing or substitute materials for some uses include raw limestone and caustic soda. Certain large lime consumers have their own quarries and lime kilns, but the percentage of such captive tonnage is relatively small, being only about 5.4 per cent of the total production of the industry in 1934.

The subject of prices is far too complex to admit of thorough discussion of even a few of the many factors entering into the exchange value of such a commodity as lime. The term "price" is freely used by many, but fully understood in all its aspects by few. Inasmuch as the lime industry has operated with a mandatory open price system (the filing and distribution of, and rigid adherence to, price lists, and terms and conditions of sale by each manufacturer) only under its Code of Fair Competition, which was in operation less than 20 months, precise data as to prices are not available over a period of years. The average value per ton as shown by Bureau of Mines statistics without doubt forms the most valuable guide as to long-time price trends, but such data do not shed light on the extent of, or changes in, factors such as cash discounts, quantity discounts, jobbers' or agents' allowances, consumer, truck, or plant pick-up differentials, container costs and adjustments thereof, rebates, methods of quotation, or the extent and economic importance of freight absorption, crosshauls and the relation of these to actual prices to consumers. Mere enumeration of some of the major elements correlated with price makes it readily apparent that the average net mill return is but a small segment of the whole, and while of relative or immediate importance to the manufacturer, data thereon can at best reflect only an imperfect image of what the price history actually has been and the evolution of pricing practices.



Fig. 1 shows the trends in average net mill returns (from Bureau of Mines statistics) for agricultural, building and chemical lime since 1909.

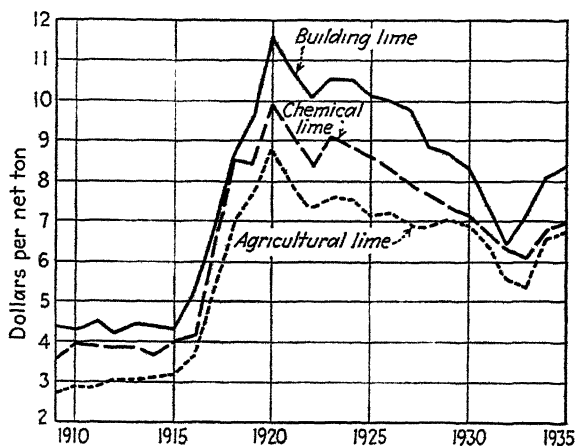


FIG. 1.—AVERAGE VALUES PER SHORT TON OF LIME.  
F.o.b. plant and exclusive of container costs. From U. S. Bureau of Mines.

Probably the most important point revealed is the relatively higher level of prices at present compared with those before the World War. Among factors contributing to this may be mentioned higher rates of wages and shorter working hours for labor, higher fuel and supply costs, demand by the trade for higher grade products necessitating more costly manufacturing processes and costs, and development of more and more packaged goods.

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## CHAPTER XXIII

### LITHIUM MINERALS

BY WALDEMAR T. SCHALLER,\* MEMBER A.I.M.E.

**L**ITHIA compounds have a place in pharmaceutical and other chemical industries and lithium minerals find use in glassmaking and ceramics. The metal, although rare, is used to a minor extent in alloys. During the nearly forty years of production of lithium minerals in the United States, the silicate mineral spodumene has continued as the chief source of raw material for the manufacture of lithium salts. Although twice as much lepidolite, a lithium mica, has been mined, most of it has been used in the making of certain types of glass. Amblygonite has been an additional but minor source of lithium compounds. Triphylite, petalite, and zinnwaldite have also been utilized in the past. All of these commercial lithium minerals contain low percentages of lithium (Table 1).

TABLE 1.—*Composition of Commercial Lithium Minerals*

Mineral	Simplified Formulas	Theoretical Content, Per Cent		Range in Commercial Mineral, Per Cent
		Lithium (Li)	Lithia (Li <sub>2</sub> O)	Lithia (Li <sub>2</sub> O)
Spodumene	LiAlSi <sub>2</sub> O <sub>6</sub> or Li <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> .4SiO <sub>2</sub>	3.73	8.03	4-8
Lepidolite . . . .	LiKAl <sub>2</sub> F <sub>2</sub> Si <sub>2</sub> O <sub>6</sub> or LiF.KF.Al <sub>2</sub> O <sub>3</sub> .3SiO <sub>2</sub>	1.90	4.09	2-4
Amblygonite . .	LiAlFPO <sub>4</sub> or 2LiF.Al <sub>2</sub> O <sub>3</sub> .P <sub>2</sub> O <sub>5</sub>	4.69	10.10	8-9
Triphylite . .	LiFePO <sub>4</sub> or Li <sub>2</sub> O.2FeO.P <sub>2</sub> O <sub>5</sub>	4.40	9.47	2-6
Petalite . .	LiAlSi <sub>4</sub> O <sub>10</sub> or Li <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> .8SiO <sub>2</sub>	2.27	4.89	2-4
Zinnwaldite . .	LiKFeAl <sub>3</sub> F <sub>3</sub> Si <sub>3</sub> O <sub>10</sub> or LiF.KF.FeO.Al <sub>2</sub> O <sub>3</sub> .3SiO <sub>2</sub>	1.58	3.40	2-3

The variation in lithia content of the commercial minerals, as mined, is due to replacement of the lithia by soda and potash, to partial alteration of the mineral to nonlithium minerals, and to the presence of impurities.

Spodumene occurs in distinct, prismatic, characteristically large crystals with a nearly square, rounded, or flat cross section. In South Dakota, crystals 20 to 30 ft. long are numerous—one measured 47 ft. long.

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\* Geologist, U. S. Geological Survey, Washington, D. C.

A diameter of 3 to 4 ft. is not rare. The commercial mineral is dull gray-white. Transparent colored varieties, such as triphane (yellow), hiddenite (green), kunzite (purple), are used as gems. The hardness of the fresh mineral is 6.5 to 7, its specific gravity is about 3.17. Spodumene is monoclinic and has the crystal habit of pyroxene, the prismatic cleavage angle being  $93^{\circ}$ . It fuses easily before the blowpipe flame, which it colors crimson, as do all the other lithium minerals.

Lepidolite commonly forms compact aggregates of glittering scales, only a fraction of an inch across, with a characteristic pink or purple tint. It may also be white, gray, yellow, purple or blue. Its hardness is about 3, and specific gravity about 2.85, with perfect micaceous cleavage. Zinnwaldite is similar in properties except for color, which is brown, yellow, violet or dark gray.

Amblygonite is generally massive, showing imperfect cleavage faces. It is white, and resembles some white feldspar, though its specific gravity (about 3.1) is higher. Its hardness is 6. Triphylite is found in cleavable masses, gray or brown, commonly stained black by separated manganese oxide, as all triphylites contain some manganese. Its hardness is 5 and specific gravity 3.5. Petalite is usually massive, resembling amblygonite and spodumene, and is white or gray.

All of the lithium minerals are typical constituents of granitic pegmatites. Zinnwaldite also occurs in veins and greisens. Most of the occurrences show evidence that they have been introduced into already formed pegmatites by hydrothermal solutions replacing earlier feldspars. In general, they occupy a somewhat central position in the pegmatites, of which they may constitute a large portion.

#### DISTRIBUTION OF DEPOSITS

Nearly all the spodumene commercially produced in the United States has come from the Black Hills of South Dakota, chiefly from the Etta mine near Keystone, Pennington County, and the Tin Mountain mine near Custer, Custer County. However, spodumene is abundant in many of the pegmatites of this region and has been produced from many other mines than those mentioned. Similarly, amblygonite is widespread in its occurrence in this region and has been mined together with the spodumene and much smaller quantities of triphylite and lepidolite. The area enclosing Keystone, Custer and Pringle would roughly outline this lithium province. The Black Hills region of South Dakota will probably continue to be the main source of lithium minerals in the future as it has been in the past.

The Stewart mine, at Pala, San Diego County, Calif., has produced large quantities of lepidolite and smaller quantities of amblygonite. Active production ceased some time ago. Similarly, the Harding mine, 13 miles east of Embudo, in northern New Mexico, produced considerable

lepidolite for a time but has not been very active in recent years. Spodumene is said to have been mined and shipped recently from a pegmatite near Warren Station, Knox County, Maine, about 60 miles northeast of Portland. In the past small quantities of spodumene and lepidolite have been produced in Maine, Massachusetts, and Connecticut. Other deposits have a potential value, although little is known of the quantity of lithium minerals present. Probably the most important of these is the group of pegmatites extending through the town of Kings Mountain, Cleveland County, N. C. Here certain zones are said to average about 20 per cent of spodumene for considerable distances.

In Colorado several thousand tons of lepidolite is estimated to occur in Gunnison County, 2 miles southwest of Ohio City. In Fremont County, a few miles west of Canon City, lepidolite and amblygonite were found. Specimens of lepidolite, amblygonite and petalite have been obtained in Fremont County, Wyoming. The extent of these undeveloped deposits in Colorado and Wyoming is not known, but nearly a carload of lepidolite is reported to be available at the Wyoming locality.

One of the most important known deposits of lithium minerals outside of the United States is in southeastern Manitoba, Canada (spodumene, amblygonite and lepidolite). Other important foreign occurrences include Utö, Sweden (petalite, lepidolite and spodumene); the Erzgebirge between Saxony, Germany, and Bohemia, Czechoslovakia (zinnwaldite); Mount Hradisko near Rožna, Moravia, Czechoslovakia (lepidolite); Montebrias and neighboring places in Creuze Department, France (amblygonite and lepidolite); the San Finx mine near Silleda, Pontevedra district, Galicia, in northwestern Spain (amblygonite); Cáceres, Cáceres district, Estremadura, in southwestern Spain (amblygonite); near Guarda, Portugal (amblygonite and lepidolite); Central Damaraland, Southwest Africa, said to be capable of furnishing from 50,000 to 60,000 short tons of lepidolite, running at least 4 per cent of  $\text{Li}_2\text{O}$ ; and Western Australia, where amblygonite, spodumene and lepidolite are reported to occur in sufficient abundance to supply the world market.

Deposits of lithium minerals in the United States could yield considerably increased production should occasion arise. The United States has produced the great bulk of lithium minerals mined and consumed, although with increased demand the deposits in Canada, Western Australia, and Southwest Africa may become important producers. The United States has both imported and exported small quantities of crude lithium minerals. There is a tariff of 25 per cent ad valorem on metallic lithium and lithium salts.

#### PRODUCTION AND MARKETING

The production of lithium minerals has always been an insignificant part of the total mineral production of any country. Domestic produc-

tion of spodumene began in South Dakota about 1898 and has continued ever since, with a total quantity of about 22,000 tons at the end of 1935. Amblygonite has been continuously mined there since about 1910, with a total of slightly more than 4000 tons. The total production of lepidolite, together with a very small quantity of amblygonite from the Stewart mine at Pala, Calif., is about 24,500 tons. The Harding mine, in New Mexico, became an active producer of lepidolite at about the time the California deposit ceased to be an important source, and has produced about 19,000 tons.

Out of a total production of about 70,000 tons of lithium minerals, spodumene from South Dakota, lepidolite from California, and lepidolite from New Mexico has each contributed about one-third. The total value at the mines of the crude lithium minerals sold (exclusive of any lithium minerals such as spodumene and tourmaline mined as gem stones) is about \$1,300,000, giving an average value of \$18.60 per ton.

Mining methods are very simple. The crystals (as of spodumene) or masses (as of lepidolite and amblygonite) are hand-picked from the broken pegmatite rock. Most of the lepidolite mined and much of the amblygonite occur in solid masses many feet thick and are simply broken down to a size convenient for handling and shipping. The crystals of spodumene from South Dakota are so immense that single crystals will yield many tons of material suitable for shipment.

No unusual preparation is required before shipment, although pieces of lepidolite containing much quartz and feldspar must be rejected. Where the mineral is to be used directly, as in glass manufacture, all iron minerals must be removed. The lepidolite from New Mexico was unusually low in iron, containing only a few hundredths of one per cent. Where the spodumene is covered with a soft, claylike material (as in South Dakota), this must first be broken off, because it contains almost no lithium.

A minimum content of lithia approximating the lower figures in the percentage range for the commercial minerals is usually specified. A price may be fixed with a bonus for lithia content exceeding the fixed minimum. For use in glass and ceramic industry, the iron content must be very low. An average price of about \$18 to \$20 a ton for lithium minerals at place of mining and shipment has held with little variation for many years. For amblygonite with a little higher content the price has at times been higher.

#### USES

The earlier use of lithium minerals was almost entirely as source material for the preparation of lithium compounds. About 20 to 25 years ago lepidolite began to be used directly as an ingredient in glassmaking. Recently renewed interest has been shown in the direct use of both

spodumene and lepidolite in the glass and ceramic industries. Lepidolite is a very good fluxing material. It also decreases the coefficient of expansion and increases the strength of glass, glazes, enamels and porcelains. Its content of lithia and fluorine, high total alkali and alumina, moderate silica and very low iron, are beneficial. The lithia makes the glass more durable and is of advantage where high electrical resistance is desired. In larger quantities, lepidolite is a good opacifier and has been used in making opal or white opaque table tops, translucent white glass for indirect lighting fixtures, ointment jars, and tops of fruit jars. Amblygonite, used directly, seems also to be beneficial in ceramics, and is reported to have been so used in glass and porcelains in France. Spodumene also is said to improve the quality of certain ceramic wares.

Minute quantities of metallic lithium impart valuable properties to certain metals (aluminum, lead, magnesium, zinc) and alloys, increasing their hardness, toughness and tensile strength. Bearing-metal alloys, copper electrodes and lead cable sheaths are so benefited. In the higher melting metals and alloys, such as iron, nickel and copper, lithium acts as a degasifier, deoxidizer, desulphurizer and general purifying agent. The lithium is generally introduced as a calcium-lithium alloy.

Manufactured salts of lithium have been put to many uses, perhaps the most widely recognized of which is in the pharmaceutical trade, large quantities being used in the preparation of mineral waters and lithia tablets. Alkaline storage batteries, using lithium hydroxide, formerly consumed large quantities. In other uses the quantity consumed for any one item may not be large, but in the aggregate amounts to an appreciable figure. The chloride and fluoride are used in welding fluxes, especially for aluminum; the borate in dental cement; the nitrite for curing meat; the hydroxide for mercerizing sulphite cellulose for rayon, and for purifying helium and other rare gases. Among many other uses of lithium salts may be mentioned that of producing red color in flares and fireworks.

A recent use of lithium chloride that may become extensive in the future is for dehumidifying air for air conditioning and industrial drying, as developed by Bichowsky. Lithium chloride is one of the most hygroscopic inorganic substances known, is nonvolatile, nontoxic, noncorrosive, and its solutions have a low freezing point.

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## CHAPTER XXIV

### MAGNESITE

BY RAYMOND E. BIRCH\*

THE mineral magnesite is the normal carbonate of magnesium,  $\text{MgCO}_3$ , composed, when pure, of 47.6 per cent  $\text{MgO}$  and 52.4 per cent  $\text{CO}_2$ . It is one of the calcite ( $\text{CaCO}_3$ ) group of rhombohedral carbonates, which also includes dolomite,  $\text{CaCO}_3\cdot\text{MgCO}_3$ , and siderite,  $\text{FeCO}_3$ . Magnesite does not form an isomorphous series with calcite or dolomite; consequently, these lime-bearing carbonates commonly present in magnesite deposits are there only as mechanical mixtures. Magnesite and iron carbonates form a continuous isomorphous series and therefore are inseparable mechanically. The variety of magnesite known as breunnerite is an isomorphous mixture of iron and magnesium carbonates.

Magnesite may be either crystalline or dense (cryptocrystalline). The former, which includes breunnerite, is often termed spathic magnesite. Crystalline magnesite varies from finely to coarsely crystalline in texture and has a hardness of  $3\frac{1}{2}$  to 4. The color is white, yellowish, blue-gray to drab, red, pink, black or mottled. It is seldom found pure but usually contains variable amounts of iron, lime, silica and sometimes manganese. The color cannot be used as an index of purity.

The cryptocrystalline variety, usually spoken of as amorphous magnesite, is perhaps the more common type, but the crystalline variety usually occurs in larger deposits. The amorphous type is fine-grained and compact, showing no cleavage. It is usually snow white but is sometimes light to pale orange-yellow or buff due to impurities. The fracture is conchoidal to uneven, and the hardness  $3\frac{1}{2}$  to 5. Silica may be present as inclusions of serpentine, quartz, chalcedony, etc. The lime and iron contents are usually low. In general, amorphous magnesite is somewhat more pure than the crystalline variety.

The specific gravity of cryptocrystalline magnesite is 2.90 to 3.00. Pure crystalline magnesite has shown a specific gravity of 3.02, but iron carbonate, commonly present, raises the specific gravity; typical figures for breunnerite are 3.08 to 3.13.

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In preparing this chapter the author has had at his disposal an unpublished article on California magnesite deposits and plant operations written by Mr. J. B. Perry, Mines Manager for the California Chemical Co. This assistance is gratefully acknowledged.

Magnesite dissociates on heating, by the release of carbon dioxide; the product is magnesia ( $\text{MgO}$ ), which develops, either simultaneously or upon further heating, a crystalline structure and is then known as periclase. The mineral periclase occurs in nature, but is rather rare and is not found in workable deposits. When dead-burned or sintered, magnesium oxide is resistant to hydration and carbonation at the temperature and pressure of the atmosphere.

#### NOMENCLATURE OF PRODUCTS

Magnesite actually refers only to the mineral  $\text{MgCO}_3$ , but common usage applies it to most of the manufactured products with a classifying prefatory word; e.g., dead-burned magnesite and caustic magnesite. For the most part these products are actually magnesia,  $\text{MgO}$ .

*Caustic Magnesite or Caustic Calcined Magnesite* is the product resulting from calcining crude magnesite at a temperature of  $700^\circ$  to  $1200^\circ$  C. About 2 to 7 per cent  $\text{CO}_2$  is left in the product, since it has been found that a calcination temperature high enough to expel all of the  $\text{CO}_2$  would impair the cementing properties of the magnesite.

*Plastic Magnesia* is caustic magnesite that is adaptable to forming magnesium oxychloride cement (Sorel cement) when mixed with magnesium chloride or magnesium sulphate solution.

*Dead-burned (or Sintered) Magnesite* is the product obtained by calcining magnesite at a temperature of  $1450^\circ$  to  $1500^\circ$  C., which dispels virtually all of the  $\text{CO}_2$  and sinters the grains to dense particles suitable for refractory purposes. It usually contains less than 0.5 per cent  $\text{CO}_2$ . At present, caustic magnesite and dead-burned magnesite consume virtually the entire production of domestic magnesite.

#### ORIGIN, OCCURRENCE, AND DISTRIBUTION OF DEPOSITS

The crystalline magnesite is generally formed by the alteration of dolomite by magnesian solutions associated with intrusions. The cryptocrystalline variety is an alteration product of serpentine or allied magnesian rocks. The amorphous variety is common in serpentine areas and occurs in fissures or crush zones mixed with serpentine and some opal or chalcedony also produced by the breakdown of the serpentine. This type of magnesite generally occurs in small deposits, as in the California coast ranges and on the Grecian island of Euboea.

\* The largest deposits of crystalline magnesite in the United States are those near Chewelah, Wash. They are replacements of dolomite of Carboniferous age, formed under the influence of later intrusives. Probably the largest deposits in the world (also crystalline) are those at Veitsch in Styria, Austria, and in Manchukuo, near Niu-hsinshan.<sup>10</sup> The Austrian

<sup>10</sup> References are at the end of the chapter.

deposit is a replacement of dolomite formed following the intrusion of porphyry and other acidic and basic rocks.

In the United States magnesite is being mined only in California and Washington. In other parts of the United States, notably Nevada, magnesite occurrences are reported, but these are not at present of commercial importance.

In Canada the only mining operation is that in the Grenville district of Quebec, but deposits are also reported in British Columbia. The Grenville magnesite is characterized by a high lime content.

The U. S. S. R. is at present the world's largest magnesite producer. Before the war Russia operated the Sutkin mines, in the Urals, but the production was insufficient for her needs. Other mines have since been opened and the total production has grown so that now the country provides sizable exports.

Viewed as to their long-time influence on the world magnesite industry the most important deposits have been those of central Europe. This magnesite is of the crystalline breunnerite type and was formed by the replacement of limestone by the action of ascending aqueous solutions carrying magnesium and iron compounds. Of the many deposits of this type that exist in a belt through central Europe, about a dozen are large enough to be workable. The narrow belt in which they occur extends westward from Semmering, Austria, the most important occurrences being at Semmering, Veitsch, Brietenau, Trieben, Radenthein and Dienten. The deposits are lenses, of which by far the most celebrated and most important is the one at Veitsch, near Mittendorf in Styria, Austria. The magnesite lens at Radenthein is also of considerable extent. Although this belt comprises the major part of the workable deposits of central Europe, Yugoslavia and Czechoslovakia also are producers.

Greece and British India are the only important producing countries whose major output is derived from cryptocrystalline magnesite. In British India most of the production is from the Salem district, in the Madras Presidency, but some supplies come from Mysore.<sup>14</sup> Magnesite exportations from Greece began in 1870 and that country has continued to hold an important world position in the industry. Prior to the war the output was second only to that of Austria-Hungary. A peak production was reached in 1916 at about 200,000 tons (crude rock). New factors in the market and perhaps local mining conditions have acted to prevent Greece from keeping pace with other producers in the two decades since the war. Her output is now less than that of Austria, Czechoslovakia, the United States, and the U. S. S. R.

Production from the large deposits of crystalline magnesite discovered in Manchukuo<sup>9</sup> doubled between 1931 and 1933.

## MINING METHODS

*Washington.*—The Washington magnesite occurs as huge, steeply pitching lenses in dolomite beds overlain by quartzite. Although six or seven deposits are known, only the Finch and Allen-Moss quarries are being operated. The Finch deposit is about 1500 ft. long by 200 ft. wide. Other important deposits, of which some were operated at one time, are the Red Marble, Double Eagle and Keystone.

The Finch quarry is mined from a deep, open-air pit or glory hole, from which chutes leading to a lower level discharge separately the good magnesite and the waste, which are separated on the quarry floor. After being blasted from the quarry walls, the magnesite is broken with sledges to pieces that a man can handle. Fine material, which tends to run higher in silica, is forked out. At the foot of the chutes the rock is carried by tram to the edge of the hill, where it is given a preliminary crushing before transportation to the calcining plant. Since no consequential amount of rock is stocked, the quarrying operation must be carried on daily. Two additional working levels have been opened above the main quarry floor, from which the good rock is delivered to the chutes on the floor of the main working.

*California.*—Magnesite is found in 22 California counties, but most of the deposits are either too small or too impure to be of commercial value, and several large deposits have been exhausted. At the present time it is reported that there are only three known workable deposits, which, as a result of consolidations, are all owned by the California Chemical Co.: (1) the Western mine, 32 miles south of Livermore (formerly operated by C. S. Maltby), (2) the Patterson mine, 28 miles west of Patterson and merely on the other side of the mountain from the Western mine, and (3) the Bald Eagle mine. The last named is the youngest mine, having been prospected in 1930. The two older mines are operated by running levels, stoping out the magnesite and back-filling. Aerial trams are used to deliver the rock to the crushing and calcining plant. Similar methods, modified where necessary, are used in the Bald Eagle mine. In the two older mines the magnesite occurs along fracture zones in serpentine. The width varies, the average being 8 ft. At the Bald Eagle mine the magnesite occurs in an accumulation of serpentine debris or breccia instead of in the solid serpentine. In these mines the magnesite varies from place to place, but by constantly blending material from various working faces a uniform rock is maintained.

*Foreign Countries.*—In foreign countries the methods of mining magnesite are varied with size of deposits and with labor conditions. In the thin-veined deposits of India and Greece much hand labor is used

and the mining patterns are similar to those used for many years in California. The most unique mining operation is probably that at Veitsch, Austria. The lens operated is nearly  $\frac{3}{4}$  mile long, over 1000 ft. thick. The magnesite is quarried in a series of terraces about 50 ft. apart vertically, extending from the summit of the hill to the base of the deposit 700 to 800 ft. lower. The large blocks of rock blasted from the quarry faces are broken to pieces of head size and cobbled to remove impurities such as schist, dolomite and quartz. The rock is transported by gravity planes to the calcining plant at the foot of the hill.<sup>8</sup> In addition to the magnesite obtained by these open methods, it is reported that a minor proportion of the output is now obtained from underground workings.

In Quebec, open-cut methods and stoping are used. The rock is broken down by blasting and is then sledged to one-man size. The rock is cobbled to remove excessive serpentine and some of the dolomitic material.

#### POLITICAL AND COMMERCIAL CONTROL

Since magnesite is necessary for the production of steel and nonferrous metals, abundant domestic supplies are a wartime necessity. Dead-burned magnesite was not regularly produced in the United States until the World War. Production of caustic magnesite began in California on a small scale in 1886, but all of that was consumed on the west coast. The year 1910 was the first year in which production (rock mined) reached 10,000 tons. Production of this order continued until the World War shut off supplies of dead-burned magnesite from Austria-Hungary. Production in California nearly trebled in 1915 and by 1917 reached an all-time peak of about 211,000 tons (crude basis) most of which was dead-burned. The State of Washington, which had not previously been a producer, came into production in late 1916; it produced over 100,000 tons the next year and reached its all-time peak production in 1920 at 222,000 tons (crude basis). By far the greater part of the Washington product has been shipped as dead-burned magnesite.

Because of the continued disorganization in central Europe after the war, Austrian magnesite did not again become available for several years. However, when importation again started the effect was a prostration of the domestic dead-burned magnesite industry, which did not attain a good measure of recovery for several years. No magnesite was shipped from Washington in 1921, owing in part to a decline in steel production but most largely to the low delivered price of the imported magnesite. However, under the tariff act of 1922, magnesite was removed from the free list. The tariff rates established in 1922 and the latest modifications in rates on crude and caustic magnesite are given in Table 1.

Although sold for use as dead-burned magnesite, the Canadian magnesite has not generally been so classified for tariff purposes. It was given special status under the 1936 Reciprocal Trade Agreement with

TABLE 1.—*Tariff Rates on Magnesite*

Magnesite	Tariff, Act of 1922		Tariff, 1937 Modifications	
	Per Lb.	Per Ton Equivalent	Per Lb.	Per Ton Equivalent
Crude . . .	$\frac{5}{16}$ ¢	\$ 6.25	$1\frac{5}{32}$ ¢	\$ 9.375
Caustic calcined . . . . .	$\frac{5}{8}$ ¢	\$12.50	$1\frac{5}{16}$ ¢	\$18.75
Dead-burned (not suitable for use in oxy-chloride cements)	$2\frac{3}{40}$ ¢	\$11.50	$2\frac{3}{40}$ ¢	\$11.50

Canada as "dead-burned basic refractory containing 6 per cent or more of lime and consisting chiefly of magnesia and lime." The duty was set at 27.5 per cent, which was a reduction from 30 per cent previously paid.

### PRODUCTION

The production of magnesite is discussed under various related headings throughout this chapter. The data necessary for a full view of the

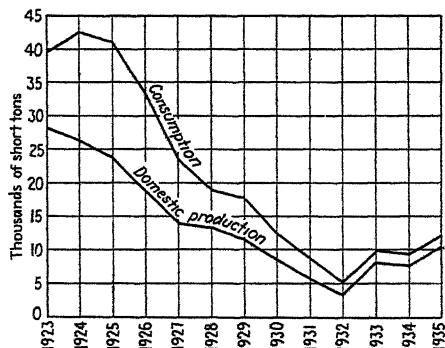


FIG. 1.—CONSUMPTION AND PRODUCTION OF CAUSTIC MAGNESITE, UNITED STATES.

picture are shown graphically in Figs. 1, 2 and 3. Fig. 1 shows the consumption of caustic magnesite in the United States (production + imports) since 1922, and the extent to which the consumption was cared for by domestic production. Fig. 2 gives similar data on dead-burned magnesite. Fig. 3 shows production of magnesite since 1920 for the

leading producing countries. The world production of magnesite has increased steadily so that the estimated production for 1934 even exceeded

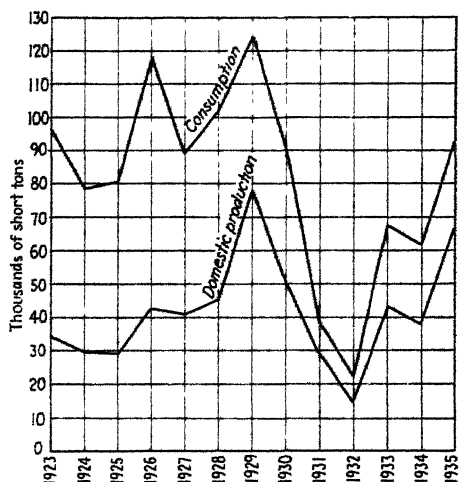


FIG. 2.—CONSUMPTION AND PRODUCTION OF DEAD-BURNED MAGNESITE, UNITED STATES.

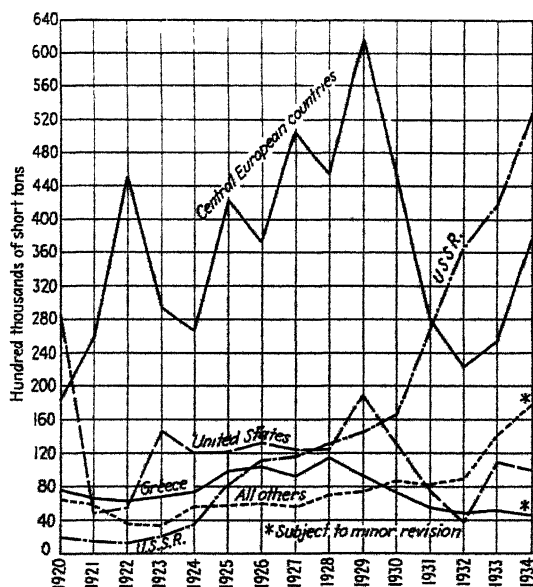


FIG. 3.—WORLD PRODUCTION OF CRUDE MAGNESITE.

the 1929 peak. The depression years, beginning in 1930, caused a precipitous fall in magnesite production in central Europe, the United



States and Greece, but the rise of the U. S. S. R. as a major producer, which occurred during these years, largely discounted these effects on the world production figures.

For the eight years ending with 1930 the central European countries produced each year approximately one-half of the world's magnesite; this share dropped during the depression years to a low at approximately 25 per cent in 1933, but has increased since then. During these same years the U. S. S. R. increased its share of the world magnesite total from less than 20 per cent to more than 40 per cent. While this would seem to indicate that the U. S. S. R. had captured a large share of the markets formerly held by the central European producers, actually this has not taken place to the degree indicated. A factor of much importance has been that the industrial nations served by Austrian and Czechoslovakian producers were using less magnesite, while the U. S. S. R. found it necessary to expand its magnesite production to take care of its own fast-growing steel industry.

#### PREPARATION FOR MARKET

*Dead-burned Magnesite.*—At the Northwest Magnesite Co., Chewelah, Wash., the operations are as follows: The mine cars dump the hand-picked rock into a large bin, from which it is fed to a jaw crusher; the 4-in. product goes to a scrubber and trommel screens, from which all material finer than  $\frac{1}{4}$  in. is washed away as waste. A second crushing operation (gyratory crushers) reduces the rock to  $2\frac{1}{2}$  in. maximum, in which form it is conveyed to the calcining plant by a 5-mile tramway.

At the calcining plant further reduction, with rolls set at  $\frac{1}{4}$  in. and  $\frac{1}{16}$  in., in series with screens, prepares the product for the rotary kiln. The kiln charge, weighed out with a small percentage of pulverized iron ore used as a sintering agent, is fed to the rotary kilns. There are six kilns,  $7\frac{1}{2}$  and 8 ft. in diameter and 125 ft. long, with burning-zone linings of magnesite brick. A burning-zone temperature of about  $1560^{\circ}$  C. ( $2840^{\circ}$  F.) is maintained during the making of dead-burned magnesite. A Cottrell treater recovers the dust from the outgoing kiln gases, part being returned to the kiln, part being utilized in by-products, and the least pure material being wasted. Each pair of kilns discharges into a rotary cooler.

The clinkered (dead-burned) product from coolers passes over a 2-in. grizzly, the oversize being put through a jaw crusher to reduce it to this size. The 2-in. product goes to  $\frac{3}{4}$ -in. impact screens, the oversize being crushed to this size and recirculated. The product ready for market as dead-burned magnesite is then put in storage. The grains are

dark brown and grade downward in size from  $\frac{3}{4}$ -in. maximum diameter. Only a minor portion is finer than 28 mesh.

*Caustic Magnesite.*—In preparing caustic magnesite at the Bald Eagle plant of the California Chemical Co. the operations are as follows: The lump rock is delivered to the crushing and calcining plant by cars and aerial tram, being fed to storage bins topped with grizzlies through which the large lumps are sledged and at which some hand sorting takes place. A primary jaw crusher with  $2\frac{1}{2}$ -in. opening receives the rock, which is then screened by a double-deck rocker screen using  $\frac{3}{4}$ -in. mesh above and  $\frac{1}{8}$ -in. mesh below. There are seven bins allotted between the three sizes: (1) pass  $\frac{1}{8}$ -in., (2) pass  $\frac{3}{4}$  in. on  $\frac{1}{8}$  in. and (3) the oversize, which is crushed to  $\frac{1}{2}$  in. by a secondary crusher of the cone type. The materials are sampled automatically on their way to storage, and the analyses are used as guides for blending. The percentage of silica, the principal impurity, is highest in the fines. From the storage bins, which have a capacity of 2000 tons and are sufficient for storage during several weeks operation, the various sizes and grades are blended and conveyed to a 60-ton bin from which the kiln is charged. An auxiliary fine-crushing unit (screens, and rolls) at the kiln-feed bin is used on grades that require exceptionally fine kiln feed.

The calcining takes place in an oil-fired rotary kiln 100 ft. long with 6-ft. inside shell diameter. The burning zone is lined with magnesite brick. The maximum temperature is about 1200° C. (2192° F.). The calcined material is discharged into a rotary cooler that contains two steel balls of 12-in. diameter, which crush the larger clinkers. The product from the kiln is stored in bins, from which it is trucked to Ingomar, Calif., for grinding or for shipment unground. Most of the caustic magnesite is shipped to the East in lump form; that treated at Ingomar is ground in a five-roll Raymond mill to 80 or 90 per cent pass 200 mesh.

#### TESTS AND SPECIFICATIONS

*Crude Magnesite.*—Crude magnesite is used in small tonnages for preparation of certain chemicals such as epsom salts, but the total annual use of crude magnesite in the United States is less than 2000 tons.

*Caustic Calcined Magnesite.*—Both chemical and physical tests may be made to test the suitability of caustic magnesite for use in oxychloride cements, but in the final analysis actual trials of its behavior in cements are necessary. The chemical analysis is valuable in showing the content of impurities and the freshness of the magnesite, since there will be an appreciable hydration and recarbonation on standing. An ignition loss of not more than 5 per cent is desired for caustic magnesite to be used in oxychloride cement. Other specifications, such as those on magnesia

and lime content, may vary somewhat with the source of the magnesite. Specifications that have been used in purchasing California caustic suitable for use in oxychloride cements are: (1) total  $\text{MgO}$ , 88 per cent minimum; (2) free  $\text{MgO}$ , 85 per cent minimum; (3) active lime, 1.5 per cent maximum; (4) water-soluble lime, 0.3 per cent maximum; (5) fineness such that a minimum of 85 per cent will pass 200 mesh. Total lime is not as important as active and water-soluble lime, since it is known that lime is harmful only when it is present in these states.

The physical tests of most value are made by preparing oxychloride cement and testing specimens for (1) setting time, (2) expansion or shrinkage and (3) strength after setting. Underburned caustic magnesite will set too rapidly and the overburned product will set too slowly and give insufficient strength. Overburning also activates the lime and tends to give the cement excessive expansion. Caustic magnesite for use in rubber need not be burned as hard as that used in oxychloride cements. Low manganese content is a requisite.

*Dead-burned Magnesite.*—Dead-burned magnesite is also subjected to both physical and chemical tests. The nature of the requirements is somewhat dependent upon the uses to which the material is put. If it is to be used as grain magnesite for building up monolithic bottoms in the basic open-hearth furnace, certain characteristics are desired that would be unnecessary in dead-burned magnesite from which brick are to be manufactured. Both services require a dense, well burned grain with a predominance of large grains.

Grains intended for the open-hearth bottom preferably contain a reasonable amount of silica (in the form of silicates), which aids the process of sintering in the hearth to form a monolithic flooring. However, excessive amounts of lime and other impurities are undesirable, since magnesium oxide is the constituent that offers greatest resistance to solution by open-hearth slag. Lime, when present in large percentages, may combine with silica to form dicalcium silicate ( $2\text{CaO} \cdot \text{SiO}_2$ ), which under certain conditions undergoes a rapid inversion which may reduce the magnesite to dust.

Iron oxide, which is preferably kept low in caustic magnesite, is a desirable constituent in dead-burned magnesite. It aids the process of dead-burning, gives a grain of greater density, and probably gives the individual grains greater powers of coherence when they are pressed together to form brick or furnace bottoms. The major tonnage of dead-burned magnesite contains 4 to 7 per cent  $\text{Fe}_2\text{O}_3$ . In the magnesite from Austria and Czechoslovakia this is a natural impurity present in the breunneritic type of crystalline magnesite found in central Europe. In the United States and Canada the iron oxide is added as kiln charge in the form of finely powdered roll scale or iron ore.

## MARKETING, USES AND PRICES

*Caustic Magnesite.*—Until several years ago most ground caustic magnesite was used in combination with magnesium chloride as oxychloride or Sorel cement but at present a larger percentage is used in making rubber. The larger outlets for oxychloride cements have been for outdoor stucco work and for flooring. A statement made in 1925 by Ralston and others<sup>15</sup> pointed out the good features of oxychloride stucco and at the same time goes a long way toward explaining what has happened to this promising market in the following decade:

Magnesia stucco has gained headway in competition with cement stucco largely because it makes a stronger, harder, and more elastic cement which adheres more firmly to wood surfaces and takes and securely holds . . . pebbles. This stucco has one disadvantage—it is not as weatherproof as cement stucco.

Owing in part to its weathering, but perhaps even more to poor application and lack of understanding of oxychloride cement, magnesite stucco fell into disrepute, so that by 1929 Portland cement had almost entirely replaced it for outside work. Fortunately, research on oxychloride cements has been intensified and there are unmistakable signs that a considerable technologic advance is now under way. It is expected that these improvements, discussed under the heading Technologic Advance, will increase the use of these cements for stucco and other outdoor applications. Assisting factors are the increased uniformity of the domestic caustic magnesite and the availability of reliable data on the compounding and use of oxychloride cements.

Fig. 1 shows the annual consumption (domestic sales plus imports) of caustic magnesite in the United States. Consumption started to fall off in 1925–1926 and the decline continued through the period of boom as well as that of depression. From 1924 to 1932 each succeeding year brought further decline in the sales of caustic magnesite. Consumption was 41,000 tons in 1924 and only 5200 tons in 1932. The market for caustic has improved considerably since the latter year. Normally, domestic producers provide about two-thirds of all caustic magnesite used in the United States. Importations at one time were, essentially all Grecian in origin, but for some years British India has been the largest source of caustic magnesite shipped to the United States. The Indian caustic is mostly in lump form, whereas that from other sources is generally ground. The most important sources of ground caustic magnesite are the Netherlands (calcined Grecian magnesite) and Yugoslavia.

While the use of oxychloride cement for stucco has decreased, its use for flooring (in which it is valued for resiliency) and wallboard apparently have increased. Other uses are found in making certain magnesium

salts, and as heat insulation, but its most important present use is as a chemical accelerator in rubber. It is reported that the amount of caustic magnesite used by the rubber industry in the United States is now several times as great as the total used in oxychloride cements.

Caustic magnesite is produced in both California and Washington, California accounting for the major part of the sales. Most caustic magnesite for eastern markets is shipped as lumps to eastern grinding plants, since this minimizes the amount of hydration occurring in transit.

*Dead-burned Magnesite.*—Dead-burned magnesite is used almost entirely for metallurgical purposes (see chapter XXXVI). There is a correlation between domestic steel production and the consumption of dead-burned magnesite in the United States. Consequently, use (apparent new supply) of dead-burned magnesite dropped precipitously from a 1929 peak of 125,100 short tons to only 22,500 tons in 1932. A substantial recovery has since been made. Viewed as a world industry, the producing capacity is considerably in excess of consumer needs for dead-burned magnesite. This situation has arisen from wartime enlargement of capacity. Dead-burned magnesite is produced in Washington and in California, but of their total output a great preponderance is shipped from Washington.

*Price.*—Although little crude magnesite is sold, the price range is of interest as reflecting mining costs. Since 1920 the market price f.o.b. west-coast mines has ranged from \$11 to \$14 per short ton. In the same period the price of the best grade of domestic ground caustic magnesite at the mines has ranged from about \$38 to \$47.50 per short ton. Since the passage of the Tariff Act of 1922, the average prices for this type of caustic have tended to show less fluctuation. A typical present price and for the decade ending with 1935 is \$40. Other grades of caustic magnesite, depending on purity and fineness of grind, sell for several dollars less per ton. The price of dead-burned magnesite (per ton f.o.b. Chewelah, Wash.) tended to fall steadily from the 1921 figure of \$35.50 to \$22 maintained from 1930 to 1936 inclusive. The present (1937) quotation is \$25 per short ton. Ordinary California dead-burned magnesite is normally priced at \$3 to \$8 more per short ton (f.o.b. California calcining plants) than Washington magnesite. Special products requiring modified manufacturing treatment are sometimes sold at higher prices.

#### TECHNOLOGIC ADVANCE

The purpose of the discussion under this heading is to call attention to some developments that may influence the future of the magnesite industry.

*Brucite.*—Brucite is magnesium hydrate,  $\text{MgO} \cdot \text{H}_2\text{O}$ . It is commonly found associated with magnesite deposits, in Washington and elsewhere.

In 1923 a deposit of brucite of considerable extent was found in Nevada, near Luning, Nye County. Within the last year, shipments of the crude rock to the East have begun. It is being calcined for sale in competition with other basic refractory grains.

*Magnesium Metal.*—The apparent production of magnesium in the United States increased from a predepression peak of about 450 tons in 1929 to more than 2000 tons in 1935. This industry is expanding rapidly in other industrial nations also. The Dow Chemical Co. is the sole producer of magnesium in the United States. Its operation at Midland, Mich., utilizes an electrolysis of magnesium chloride obtained from deep brine wells.

It is too early to predict to what extent magnesite may come to be used as the crude material for producing the metal. In the United States the cost of transporting the magnesite to the East would be disadvantageous, although the utilization of Washington magnesite in producing magnesium is the subject of a study being conducted at the State College of Washington.<sup>5</sup>

In Austria, magnesium is being produced from magnesite on a commercial scale. It is reported that calcined magnesia is reduced with coal in an electric furnace at about 2200° to 2300° C., the metal being vaporized and later condensed by sudden chilling. Plans have also been made to utilize Manchurian magnesite in making magnesium. A plant for this purpose has been built in the City of Ubé, Yamaguchi prefecture, western Japan.<sup>9</sup>

Perhaps it may be said that the utilization of magnesite in making magnesium in the United States and Canada will depend upon many factors, including: (1) the availability of a practical process, (2) the patent situation, which could conceivably present obstructions, (3) the ability to compete with processes utilizing salt brines and possibly dolomite. The production of magnesium from dolomite has recently been projected in Great Britain, but, so far as is known, the utilization of magnesite or dolomite in producing magnesium is not now being actively contemplated except in Europe.

*Magnesia from Sea Water.*—For several years the California Chemical Co. has operated a small pilot plant at Newark, Calif., which has successfully recovered magnesium chloride from sea-water bitterns. Caustic and dead-burned "magnesite" have also been produced and have been used successfully on a commercial scale. A larger plant, to have a daily production capacity of about 50 tons of magnesium oxide, is under construction. The Marine Chemicals Co. has also recovered magnesium salts from sea water near San Francisco.

*Development of Caustic Magnesite.*—Almost certainly, the decline in the use of oxychloride cements, which began a decade ago, has now

reached its end, and it is felt that there will be a steady but perhaps slow advance based on the unmistakable advance in technology. A present view of the situation shows that reliable data on compounding and using oxychloride cements and their behavior are now available. Work done by the Dow Chemical Co.<sup>18</sup> is given much credit for the orderliness that has been introduced into data bearing on these cements.

There has been no decline in research directed toward the improvement of oxychloride cement. That even the old problem of weatherability is solvable is indicated by recently announced results of the researches of Dean S. Hubbell and associates at Mellon Institute.<sup>7</sup> By the inclusion of fine copper powder in the oxychloride cement, resistance to weathering is greatly increased, and marked improvement is noted in strength, abrasion resistance, expansion characteristics, and other properties. This new type of oxychloride cement can be used indoors or out, for such exacting purposes as resurfacing worn concrete and for thin floor veneering, in which its remarkable adhering property is of service.

*Magnesite Beneficiation.*—There is a possibility that the application of improved separations methods to the purification of magnesite may alter to a certain extent the outlook for domestic magnesite. In present mining operations much of the rock wasted because of failure to meet analysis specifications needs only a very slight improvement to render it usable. It is probable therefore that the adoption of improved gravity separation methods or flotation for magnesite rock will increase the domestic reserves. Separation of magnesite from dolomite to reduce the lime content is an extremely difficult problem, as indicated by the prolonged research on this phase of the development of Canadian magnesite products. Removal of serpentine and quartz should be less difficult.

Since magnesite is found as an accessory mineral in mineral deposits in the eastern United States, there is a possibility that some day it may be produced close to the markets of this area. Recent tests by the United States Bureau of Mines<sup>15</sup> indicate that a flotation operation used by the Eastern Magnesia Talc Co. in beneficiating Vermont talc gives tailings that may be salable as magnesite. These tailings carry 85 to 90 per cent magnesite.

*Magnesite Refractories.*—A number of years ago magnesite refractories were definitely known to be losing markets to chrome refractories, particularly in the open-hearth steel furnace. While this trend may or may not be continuing, magnesite refractories have certainly widened their market in a number of other industries (see chapter XXXVI). The position of magnesite as a refractory is modified by the technologic advances, which have added new basic to neutral refractories that contain magnesite as

a minor constituent. A few years ago only chrome brick and magnesite brick were available as basic to neutral refractories for use in the steel plant; today the situation is greatly changed; the refractories manufacturers have learned that some unique and unanticipated improvements are obtainable by blending chrome and magnesite and by the use of olivine, a newcomer to the list of industrial minerals. In these new blended refractories magnesite is the one essential constituent; when fine-ground it reacts with the low-melting magnesium silicate impurities of the serpentine type in the chrome ore and olivine to form the highly refractory magnesium orthosilicate forsterite ( $2\text{MgO} \cdot \text{SiO}_2$ ). Refractories made in this way are better able to support load at furnace temperatures. A survey of the available basic to neutral refractories shows that there are at present five distinct types of brick, of which four types contain some magnesite. For this reason it may be said that although magnesite brick may have suffered some losses to other refractories in recent years, the use of magnesite as a refractory raw material has probably gained.

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## CHAPTER XXV

### MANGANESE

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**M**ANGANESE ore is a true jack-of-all-trades among industrial minerals, its only possible rival being iron ore. It is used in porcelain enamel, dry batteries, building brick, glazed pottery, plastics, welding rod, chemicals, varnish and floor tile. You may be reading this through spectacles decolorized with manganese and the light bulb above you also contains some of this element. The steel industry is the largest consumer, taking more than 90 per cent of the output, but this chapter will be confined largely to uses of manganese outside of the metallurgical field.

#### ORE OCCURRENCE

Practically all of the manganese of commerce is obtained from two minerals with odd names, psilomelane and pyrolusite. Both are oxides and the two minerals are often intimately mixed in the same ore bed. Pyrolusite is a black mineral, which is usually sooty and soft enough to soil the fingers when handled. Its chemical composition is manganese dioxide, but like all ores it contains some impurities such as silica, iron and lime. Psilomelane is essentially manganese dioxide and manganese monoxide with some combined water, barium oxide and small amounts of iron. It is harder than pyrolusite and brownish black or the color of gun metal. Nearly all commercial manganese deposits are of secondary formation; that is, they are concentrated from manganese minerals more sparsely distributed in rocks. The minerals are usually found in the form of irregular masses, which occur as ore beds of varying thickness and purity. Some manganese occurs in soft gravel or clay formations, but in other places the mineral is found in the form of veins and rounded nodules in limestone and hard rock. The U. S. Geological Survey suggests the following classifications: (1) stratiform masses, (2) veins and breccia filling, (3) irregular masses, (4) aggregates in clay or weathered rock, (5) unclassified deposits. Typical analyses are given in Table 1.

Manganese is widely scattered in all parts of the world in many types of deposits, but relatively few can be mined profitably. The three most important producers at the present time are Russia, African Gold Coast

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TABLE 1.—*Analyses of Manganese Ore*

Constituents	Caucasian	Indian	Gold Coast	Montana
MnO <sub>2</sub> , per cent	87.0	86.75	85.14	70.0
Fe, per cent	0.8	0.72	1.50	2.25
Al <sub>2</sub> O <sub>3</sub> , per cent	1.50	1.11	2.32	1.25
SiO <sub>2</sub> , per cent	5.00	3.24	2.01	12.0

and British India. These major sources have immense reserves and could easily supply the entire amount consumed in the world. Another important factor in the world market is Brazil, which usually mines about 8 or 9 per cent of the total. The world production of manganese averaged just under three million tons yearly for the nearly normal five-year period from 1927 to 1931. During the same five years the United States produced only 1.7 per cent of the world output, in spite of the large market here and a protective tariff. Manganese happens to be one of the very few essential raw materials for which we must depend upon foreign sources of supply. The writer believes that domestic production may increase somewhat in the next few years, because of the recent commercial development of flotation processes that may be used to beneficiate some low-grade American deposits of rather large extent.

#### PREPARATION FOR MARKET

The lump ore intended for chemical or ceramic use is generally hand-sorted either at the mines or at the grinding plant. Pieces of limestone, clay and limonite are carefully removed either by hand or by one of the customary concentration methods. Each lot of ore is analyzed before it is processed. The manganese first passes through an oil-fired rotary drier and the lumps are then reduced to  $\frac{3}{4}$ -in. size in a jaw crusher. Some trades, such as battery and brick manufactures, require a granular product, and for these consumers the manganese is further crushed in roll-type grinding units and then screened to exact specifications. Other users prefer a finely powdered, air-floated material. This is produced in ball-race mills connected in closed circuit with an air classifier. The final product is so fine that 80 or 90 per cent passes a 325-mesh screen.

#### COMMERCIAL USES

*Dry Batteries.*—The battery industry in the United States uses thousands of tons of manganese annually as a depolarizer in dry cells. Montana produces an ore suitable for this purpose and in 1935 the Bureau of Mines reported that 7264 tons was shipped to the battery trade from Montana mines. Puerto Rico supplied 3358 tons to this industry in 1935 and Africa much larger tonnages. No one seems to know exactly why dry cells made with certain manganese ores have good "shelf life" whereas

batteries made with higher dioxide ores may be quite unsatisfactory. Apparently the best results are obtained from manganese with a high percentage of combined water and a low lime content. It is also believed that ores containing very little copper, nickel and other heavy metals are best for battery use.

*Manganese Sulphate.*—There are several manufacturers of this salt in the United States and almost every one of them has developed a different method of production. In one of the older methods finely powdered ore is mixed with concentrated sulphuric acid to form a stiff paste. This is heated in a furnace and the soluble manganese sulphate is leached out and evaporated. This salt serves as a starting point in the manufacture of manganese carbonate, manganese phosphate and manganese driers.

*Fertilizers.*—There has been a steadily growing use for manganese sulphate as a fertilizer in limestone soils. A few years ago the Florida Agricultural Experiment Station conducted some extremely interesting experiments with this salt. They found that truck crops such as tomatoes, beans, potatoes and corn failed to fully mature in the high-lime Glade soils when treated only with commercial fertilizers, but the same soils produced healthy, profitable crops when a definite percentage of manganese sulphate was added. The results of these Florida experiments read like a "before-and-after" advertisement for hair tonic. For example, 50 lb. of manganese sulphate per acre increased the yield of snap beans 400 per cent and the quality of the beans was much better. With Irish potatoes the yield was two to three times as great as that secured without manganese. So far the use of manganese sulphate in agriculture has been confined largely to southeast Texas and Florida, but it is now being used in other states as an addition mineral in fertilizers for tobacco.

*Manganese Chloride.*—This is a rose red, hygroscopic, crystalline salt. It is soluble in water and picks up moisture from the atmosphere with such disconcerting ease that it must be kept well covered. It is not manufactured in the United States because it can be bought more cheaply in Europe, where it is a by-product of some of the old chlorine plants. Manganese ore is such a strong oxidizing agent that it will decompose hydrochloric acid and evolve chlorine. This property is the basis on which these old European chlorine plants operate. The simplified reaction is as follows:



The by-product manganese chloride is used in producing black flashed brick and in making some manganese driers.

*Manganese Driers.*—Manganese dioxide and certain manganese salts are used as driers in varnish, japan and printing ink. Linseed oil and other oils used in varnish dry by oxidation and the rate of oxygen absorp-

tion of the oils is increased by the addition of manganese. It is such a powerful drier that 0.25 per cent is usually the maximum amount added to linseed oil. The manganese can be introduced in the varnish as the linoleate, the resinate, the borate, the oxalate, the sulphate or the precipitated dioxide. In many of the old types of japan and varnish, finely powdered manganese ore is used.

*Hydroquinone*.—Manganese dioxide is employed as an oxidizing agent in the manufacture of hydroquinone. For this use the ore must be very finely ground and must have a physical structure that permits rapid reactions and good solubility in sulphuric acid. Hydroquinone is principally used as a photographic developer for both still and moving picture film. It has minor uses as a powerful medicinal antiseptic.

*Potassium Permanganate*.—This purple crystalline salt is another of the products made from manganese ore. It is soluble in water, producing a solution with an intense reddish violet color. Potassium permanganate is a strong oxidizing agent for organic and inorganic substances. Its chemical uses cover a wide range from dry-cleaning shops to lithopone and electrolytic-zinc plants. Moderately large quantities are sold for use as a disinfectant.

*Glass*.—Manganese imparts a lavender tint to glass, which neutralizes the yellowish green color due to traces of iron in the sand and other batch materials. In some cases, 1 lb. of manganese dioxide is sufficient to decolorize a 2500-lb. batch. However, glass decolorized with manganese has the curious property of sometimes changing to pink on long exposure to light. For this reason, during the last 20 years it has been largely replaced by selenium in glass decolorizing. In optical and lamp glass it is still frequently used. If excess manganese is added, the glass becomes deep purple or black. Theorists say that it is impossible to produce a true black glass or black glaze, but a mixture of manganese and either iron or chrome oxide is so nearly perfect that all but the theorists are satisfied. The black glass used in stemware, in ornamental objects and in bottles derives its color from manganese. Manganese also colors the flat or plate glass used in black table tops, store fronts and wall panels.

*Porcelain Enamel*.—Some shades of dry-process enamel bathtubs contain small quantities of manganese dioxide and it is employed to a larger extent in the wet-process enamel applied on teakettles, refrigerators and enameled signs. It is particularly useful in ground coat enamels, as a combination of cobalt and manganese has the peculiar and valuable property of bonding with the steel base. A typical ground coat may contain 0.5 per cent cobalt and 1.5 per cent manganese.

*Gray Brick*.—Manganese is the colorant used to produce the beautiful gray speckled brick for public buildings, hotels, schools and homes. For this purpose a metallurgical type of manganese ore is satisfactory but it must be granulated and screened to exact specifications. Slight varia-

tions in screen size show up plainly after the brick are burned, because the particles of manganese fuse to several times their original size. Brick manufacturers usually add from 25 to 60 lb. of manganese per thousand brick. It is employed primarily to neutralize the yellowish buff color of the clay. The secondary purpose is to provide specks, which give the brick a lively appearance. Manganese was first used in brick some 40 years ago by a small plant at Union Furnace, Ohio. Since that time there has been no marked change in the process.

*Black Brick.*—The best black brick are produced with manganese. Sometimes the entire clay body is colored by the addition of 80 to 120 lb. of finely powdered manganese per thousand brick. This method is rather expensive and therefore the majority of the manufacturers flash their brick. A good, blue-black flash can be put on the surface of the brick by throwing manganese in the fireboxes at the end of the burn. The kiln is first given a heavy smoke flash for several hours. The manganese dioxide is mixed with common salt and 8 to 10 lb. is spread on each fire. A half hour later a second shovelful is added to the fires and sometimes a third round is used. During the flashing period the kiln should be in a moderately reducing condition and the temperature should be at least 1850° F. After the flash the kiln is cooled rather rapidly. The best results are obtained with air-floated manganese ore, as this is more thoroughly circulated in the kiln than a coarser product and consequently produces a larger percentage of flashed brick. Manganese chloride is also used to produce a fine jet black flash. It volatilizes easily on a hot fire and can often be used successfully on brick that will not withstand the higher temperatures necessary with manganese dioxide.

*Pottery and Tile.*—The coloring value and stability of manganese at all firing temperatures were discovered and appreciated many years ago by the pottery trade. It imparts a strong brown color to ceramic glazes. For example, the chocolate brown glaze on porcelain insulators for high-tension lines is often produced by a mixture of manganese, chrome and red iron oxides. It is also the principal colorant in brown earthenware jugs and garden pottery. Manganese is such a reliable raw material that it is hard to find a ceramic manufacturer that does not employ it to some extent either in the glaze or in the body. For the latter, its application is similar to its use in gray and black brick. Plants that manufacture terra cotta and floor tile mix carefully granulated manganese with the clay to produce a gray speckled effect. When black floor tile are desired, it is possible to get a fairly good shade with red clay by the addition of 2 to 4 per cent finely ground manganese ore. Using buff clay, a good brown can be obtained with 2 per cent manganese chloride.

*Welding Rod.*—Manufacturers of coated welding rod use a wide variety of materials in the coating composition. Two of the common ingredients in a coating formula are manganese carbonate and manganese dioxide.

Their principal purpose is to produce a brittle, fluid slag, and to replace the manganese that is volatilized in the arc.

*Minor Uses.*—From a chemical standpoint, manganese is remarkable in that it shows valences of two, three, four, six and seven in different compounds. It forms a wide variety of salts, some of which have small commercial uses. For example, barium manganate is a green pigment, manganese arsenate is an insecticide, and manganese phosphate is a protective coating for steel.

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## CHAPTER XXVI

### MICA

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THE tonnage of mica produced annually is small, but its special properties make it essential with electrical industries and important to many others. The group of mica minerals includes a number of species, differing widely in composition. Essentially, the micas are silicates of alumina with potash and hydroxyl, but numerous other elements often enter into their composition, so that precise formulas are often difficult to assign. Iron, both ferrous and ferric, is commonly present, either as an essential constituent or as an impurity. Other characteristic elements include magnesium, sodium and lithium. Any one of such elements present as an essential component gives its name to the particular species; e.g., potash mica (muscovite), magnesia mica (phlogopite), lithia mica (lepidolite), etc. One element, however, may replace another in part, so that iron-rich phlogopites grade towards biotite with a corresponding approach to the latter in black color, brittleness, lack of ready cleavage, etc. Fluorine is prominent in some varieties, and, rarely, barium, manganese, titanium, chromium, rubidium, caesium, vanadium, and other elements, may be present in traces. The source of the titanium found in some phlogopites is thought to be microscopic needles of the mineral rutile ( $\text{TiO}_2$ ). Standard works on mineralogy describe the various varieties and subvarieties of the mica group. From the commercial angle, muscovite, phlogopite and biotite are the ones of primary importance.

### COMPOSITION

Muscovite—potash mica; theoretical formula‡  $\text{H}_2\text{KAl}_3(\text{SiO}_4)_3$ —is sometimes termed in the trade “white mica,” to differentiate it from “amber mica” (phlogopite). This is not exact, because muscovite crystals, or “books,” usually are of a greenish, brown or reddish shade although almost all micas, except biotite, yield transparent, colorless

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‡ Formulas for the different micas are listed from Dana, Ed. 6, with the exception of vermiculite, of which the composition has recently been determined by J. W. Gruner [*Amer. Mineralogist* (Dec. 1934) 19, 557-575].



sheets when split into thin films. The red muscovites are termed in the trade "ruby mica." Muscovite frequently has black and red spots or blotches, because of extremely thin films of iron oxide between the laminae. It is the most valuable industrially of all the micas, and the variety most extensively mined throughout the world. Fuchsite, a variety of muscovite containing chromium, is an attractive emerald green. Fuchsite schist makes a handsome ornamental stone when polished, and in crushed form has been employed for decorative effect in stucco, artificial stone, et cetera.

Phlogopite—magnesia mica; theoretical formula  $(\text{H}, \text{K}, \text{Mg}, \text{F})_3 \text{Mg}_3 \text{Al}(\text{SiO}_4)_3$ —also is termed in the trade "amber mica," from its prevailing color, which ranges from pale yellow to blackish brown. Color plays an important part in the commercial grading of phlogopite mica, such terms as "light silver amber," "dark silver amber," "wine amber," etc., being used to designate the shades. Phlogopite is less transparent than muscovite, exhibiting a faint cloudiness or milkiness even in very thin films.

In biotite—magnesia-iron mica; theoretical formula  $(\text{H}, \text{K})_2(\text{Mg}, \text{Fe})_2\text{Al}_2(\text{SiO}_4)_3$ —the iron content is variable, and with decreasing amount of iron biotite grades over toward phlogopite. Biotite with a high content of ferric iron is called lepidomelane.

Vermiculite—jefferisite, theoretical formula  $(\text{OH})_2(\text{Mg}, \text{Fe})_3(\text{Si}, \text{Al}, \text{Fe})_4\text{O}_{10} \cdot 4\text{H}_2\text{O}$ —may be regarded as hydrated biotite (in part "hydrobiotite") that has undergone alteration by hydrothermal agencies. It possesses the peculiar property of exfoliating to a remarkable degree when strongly heated, because of the formation of steam between the layers. Regarded as a mineralogical curiosity until about ten years ago, vermiculite has now become of considerable industrial importance for its valuable sound-insulating and heat-insulating properties.

Lepidolite—lithia mica; theoretical formula  $\text{KL}[(\text{Al}(\text{OH}, \text{F})_2)\text{Al}(\text{SiO}_3)_2]$ —has a variable ratio of fluorine and hydroxyl. It often contains the rare elements rubidium and caesium. It has a characteristic lilac-rose to violet-purple color and is rarely found in large, clear sheets; usually it is a compact aggregate of fine flakes. The uses of lepidolite are limited: most of the comparatively small production is employed in the glass industry for the manufacture of special types of glass, and it is employed to a limited extent as a source of lithia in the manufacture of lithium salts.

Zinnwaldite—lithia-iron mica; theoretical formula  $(\text{K}, \text{Li})_3\text{FeAl}_3\text{Si}_5\text{O}_{16}(\text{OH}, \text{F})_2$ —is one of the rarer micas, found in some lithia pegmatites and in tin-bearing greisens. It seldom occurs in large sheets; usually in small crystals and flakes. It has a gray to smoky brown color. Its only use is as a source of lithia, for which purpose it has been employed in Germany in recent years in the manufacture of lithium salts and metal.

Roscoelite—vanadium mica; theoretical formula  $\text{H}_3\text{K}(\text{Mg}, \text{Fe})(\text{Al}, \text{V})_4(\text{SiO}_3)_{12}$ —is a rare mica, found as fine flakes in certain sandstones in Colo-

rado, Utah and Arizona, these being its only important occurrences. Its only use is as a source of vanadium.

### PROPERTIES

With the exception of lepidolite and zinnwaldite, both lithium micas, and roscoelite, vanadium mica, which are only used in industry as sources of the elements indicated, the mica of commerce owes its usefulness to its form and physical properties. All micas possess highly perfect basal cleavage, enabling the natural crystals to be split readily into thin sheets or films. Perfect crystals, free from crystallographic or mechanical imperfections, can be made to yield films whose thinness is limited only by the mechanical strength of the mica and the dexterity of the operator. In regular practice, muscovite and phlogopite films (splittings) are produced in quantity to meet specifications calling for a thickness of not over one one-thousandth of an inch (one mil). The dark colored, iron-rich micas do not split nearly as well as the lighter colored varieties. Crystallographic imperfections, due to twinning, intergrowth or distortion, seriously affect splitting quality, as do also foreign mineral inclusions within the crystals and, in flattened form, between the laminae. A large proportion of the mica mined is useless as sheet mica for one or other of the causes mentioned above, and can be utilized only as grinding scrap for the production of mica powder.

Mica is variable in its hardness, commonly ranging from 2 to 2.5 for the softer phlogopites to 3 to 3.5 for the iron-rich varieties, biotite, etc. The specific gravity of the various members of the group ranges from 2.75 to 3.2. All of the micas crystallize in the monoclinic system, commonly in the form of flattened, six-sided crystals: with elongation in the direction of the vertical axis, an approach to hexagonal, prismatic form is achieved, usually with a more or less pronounced taper towards the basal termination. Good crystal form is rare, however, especially with larger crystals, and mica books are more often rather ragged and irregular in outline.

All micas yield water when strongly heated, the moisture content being usually around 5 per cent. This is probably water of constitution, and the micas are therefore not strictly hydrous silicates. An exception to this is vermiculite, which contains from 11 to 21 per cent water, and is definitely hydrated. Muscovite commences to lose water rapidly at 700° C., but phlogopite remains practically unaffected at temperatures below 1000° C.

In color, mica ranges through practically the entire scale, from water-clear to dense black and opaque. The color depth naturally varies with the thickness of the sheets, and all but the blackest micas are almost colorless and transparent when split into thin films.

Mica has low thermal conductivity (3.6) and high dielectric strength, and it is these properties, coupled with comparatively high heat resistance, toughness, flexibility, resilience, and ability to split into thin sheets, that give the varieties muscovite and phlogopite their outstanding value as electrical insulating materials. According to Monkhouse,<sup>6</sup> the dielectric strength in kilovolts per mil, at 20° C., ranges from 3.25 to 6.25 for muscovite, and from 3.7 to 4.2 for phlogopite. Horton, in a recent report of the U. S. Bureau of Mines,<sup>14</sup> gives the results of an extensive series of tests on a number of micas from various parts of the world. These tests show dielectric strengths of from 3.3 to 8.5 for muscovite and from 4.3 to 5.9 for phlogopite. The same report gives the dielectric constant of muscovite as from 6.93 to 8.83, and of phlogopite from 5.41 to 7.07; the power factor of muscovite ranging from 0.014 to 8.36, and of phlogopite from 0.38 to 7.12. While fusible at high temperatures, both varieties will withstand considerable heat without important change. Muscovite is less heat-resistant than phlogopite, however, and can be used for electrical purposes only where the temperatures involved will not exceed 550° C., whereas the best phlogopites will withstand temperatures up to 1000° C. No other material, natural or artificial, has ever been found that combines in itself all the desirable properties possessed by these two varieties of mica for the wide range of duty demanded in the electrical industry, and it seems probable that mica will long continue to be an essential industrial mineral in this field. Considerable attention has been given to attempts to develop substitutes for mica for electrical insulation, but nothing entirely satisfactory has yet been found. Such materials as "Micarta," "Pertinax," and other similar proprietary products, have been placed on the market and are used for certain purposes. They are all synthetic varnished paper or varnished fabric.

#### ORIGIN AND MODE OF OCCURRENCE

Mica occurs both as a primary and as a secondary mineral, but the valuable sheet mica of commerce is of primary origin. The two most common micas, muscovite and biotite, in the form of small flakes, are essential rock-forming constituents, and, as such, are of world-wide distribution in granites, gneisses, schists, and similar rocks. In the crystalline igneous rocks the mica is, of course, primary; in metamorphic rocks, it is usually a secondary mineral. Crystalline magnesian limestones or dolomites often contain small flakes of secondary phlogopite. Secondary muscovite is often sericite and tends to have a fibrous form and to be lower in potash than the normal mineral. It is sericite, common in many ore-bearing rocks, that has recently been suggested as the chief cause of silicosis in miners.

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<sup>6</sup> References are at the end of the chapter.

Primary\* sheet mica occurs solely in rocks known as pegmatites. These are dike-like bodies, often large, intruded into older rocks from deeper seated massifs. While the mineral composition of pegmatites is essentially the same as that of the parent rock from which they are derived, they are distinguished by the often enormous scale on which their component minerals have crystallized.

The world's supply of sheet muscovite mica is derived from acid pegmatites having the same mineral composition as ordinary granite, feldspar, quartz and mica. The large muscovite crystals, or "books," as they are termed by the miners, may attain a diameter of several feet, though such dimensions are comparatively rare and sheets 12 to 18 in. across represent the largest sizes normally found. Pegmatites are widely distributed throughout the world, and many countries have contributed to the world supply of sheet muscovite. Biotite mica, useless except as grinding scrap, is also sometimes found in granite pegmatites, occasionally associated with muscovite and more rarely intergrown with the latter. Muscovite is generally rare or absent in biotite-rich dikes.

Lepidolite, or lithia mica, is confined to granite pegmatites whose source magma was rich in lithia, and such pegmatites are distinctly regional in their distribution. While not exactly rare, their known occurrence is confined to certain limited areas; e.g.: the Black Hills region, in South Dakota; the Pala district, in San Diego County, California; the Embudo district, New Mexico; and the Winnipeg River region, in southeastern Manitoba, to mention the principal localities on the American continent. In Southern Rhodesia, Southwest Africa, Portugal and France, there are also important occurrences of lepidolite.

Phlogopite is restricted to basic pegmatites (pyroxenites), composed chiefly of the mineral pyroxene, and essentially quartz-free. Such dikes often are rich in phosphorus and fluorine in the form of apatite (fluorophosphate of lime), and many of the Canadian deposits have been worked both for their mica and phosphate content. The occurrence of phlogopite-pyroxenite pegmatites is distinctly regional, being confined to areas underlain by large batholithic masses of basic, pyroxenic rocks. The most important of such known areas lies in southwestern Quebec and southeastern Ontario, which for many years was the only important world source of phlogopite mica. More recently, another such area has been discovered in Madagascar, and that country now produces important quantities of this type of mica. Small amounts have also been furnished by Ceylon, India, Russia and Korea.

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\* The word "primary" is used here in a relative rather than an absolute sense. In recent years, the study of pegmatites has led to the belief that many pegmatitic minerals are the result of slow and progressive changes taking place within the original dike masses through the agency of repeated surges or flows of ascending solutions released from the parent magma during its final stages of cooling and consolidation.

Vermiculite (jefferisite or hydrobiotite) is regarded as a biotite, or perhaps phlogopite, that has suffered a pronounced degree of alteration (hydration) by natural hydrothermal agencies. It is a comparatively rare species of mica, known only from a few scattered localities. The most important known occurrences are in the United States, the largest deposit being situated near Libby, Mont.<sup>21</sup> Other deposits are reported to occur in Colorado, Wyoming, North Carolina and Georgia.<sup>23</sup> Recently, the mineral was reported to have been found in Tanganyika, Africa.

The Libby vermiculite occurs as a single huge mass or stock, the bulk of which consists of an irregular assemblage of mica crystals, ranging in size from  $\frac{1}{4}$  in. to as much as 6 in. across. Most of the crystals are 1 in. and under. The deposit is cut by numerous rather narrow dikes of an igneous intrusive rock, and to these intrusions is probably due the alteration of the original biotite, which apparently represents an unusually rich concentration of mica within a partly serpentinized basic intrusive stock (pyroxenite, dunite or amphibolite). Other vermiculite deposits probably are similar in general origin.

The remaining varieties of mica are of comparatively small economic importance. Zinnwaldite, the lithia-iron mica, is occasionally found in lithia pegmatites, usually in rather small crystals or aggregates of crystals. A recent paper<sup>12</sup> describes the finding of unusually large zinnwaldite crystals at a mine in the Amelia district, Virginia; the crystals attain a diameter of nearly 9 in. and the sheets are clear and transparent, brown in color, resembling muscovite. The only commercial use ever made of zinnwaldite is for the extraction of the contained lithia, considerable tonnages of the mineral having been taken in recent years from old tin workings in the Zinnwald-Altenberg district, in the Saxon Erzgebirge, Germany, for that purpose. The occurrence there is not in true pegmatite, but in a quartz-mica rock (greisen) resulting from the alteration of a coarse granite. Roscoelite, or vanadium mica, which contains from 1.5 to 3.5 per cent vanadium, is known only as fine, greenish scales in certain sandstones in Colorado, Utah and Arizona. It has been used on an extensive scale as a source of vanadium. Fuchsite, or chrome mica, is known only as small crystals or flakes, usually in schistose rocks, and probably is usually of secondary origin. It deserves mention here only for its handsome emerald green color, which makes the rock containing it of possible value as a decorative stone. It finds no industrial use.

#### DISTRIBUTION OF DEPOSITS

The United States possesses important reserves of sheet mica, all of the muscovite variety. Deposits are known in 15 or more states,<sup>8,14,26</sup> but the main productive areas are contained: (1) in a belt stretching from central Virginia through the western portions of North and South Carolina and Georgia, into eastern Alabama; (2) in a small region in

southwestern New Hampshire; and (3) in the southern portion of the Black Hills, South Dakota. Other muscovite-bearing states include New Mexico, Maryland, Maine, Connecticut, Colorado, Idaho, Wyoming, Texas, Nevada and California. The total production from the latter states, however, has been small, compared with that of the two main producers, North Carolina and New Hampshire. In 1934, North Carolina produced 50 per cent of the total sheet mica output of the United States, and New Hampshire, 28 per cent.\*

Of the micas other than muscovite and biotite, the United States possesses important reserves of lepidolite, or lithium mica. The principal deposits are in the southern Black Hills, South Dakota; near Embudo, New Mexico; and in San Diego County, California. The Californian deposits have been the principal source of the lepidolite mined, with a total recorded production up to 1929 of 21,875 tons. Most of the output has been used in the glass industry, as a batch ingredient for certain types of heat-resistant and opal glass.

The rare vanadium mica, roscoelite, occurs as fine flakes in sandstones near Rifle and Placerville, Colo., as well as in adjacent sections of Utah and Arizona. The deposits, associated sometimes with carnotite, have been mined on a considerable scale for their vanadium content.

The occurrence of vermiculite, or jefferisite, is restricted to comparatively few localities, the mineral being dependent for its origin on hydrothermal agencies which, as the result of local intrusive action, have brought about an alteration (hydration) of original biotite or phlogopite. The United States possesses the largest known deposits of this type of mica, the most important being the deposit near Libby, Mont.<sup>21</sup> This deposit is believed to be capable of supplying any prospective demand for an indefinite period. Other deposits occur in the Turret and Westcliffe districts, Colorado; near Encampment, Wyoming, and in North Carolina and Georgia.<sup>22</sup> Up to the present, few of these have been actively developed, and the Libby deposit has furnished the bulk of the production.

As already indicated, muscovite mica is a widely distributed mineral, and deposits are known in many countries. In some places it is a by-product of feldspar mining, and a considerable proportion of the American production is derived from feldspar mines.

The world is largely dependent on India for supplies of high-grade muscovite for the electrical industries, her output being far ahead of that of any other country. The principal producing mines lie in Kodarma (Hazaribagh) and Nellore, Madras, the two districts being generally referred to as the Bihar field and the Nellore field, respectively. The Bihar muscovite is characterized by a rich, ruby color, is of exceptional

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\* F. W. Horton, and B. H. Stoddard: U. S. Bur. Mines, Minerals Yearbook, 1935, 1177.

quality and is known in the trade as "Bengal ruby mica." India also produces a small amount of phlogopite mica from deposits in Travancore.

After India, Madagascar has recently ranked as the next most important mica-producing country, followed by the United States and Canada. The output of both Madagascar and Canada is almost entirely phlogopite. Korea and Ceylon appear to be the only other countries possessing phlogopite deposits, though there have been reports of occurrences of this type of mica in Russia.

Owing to the lack of precise details of the grade of product shipped, it is difficult to gather from statistical records the correct standing of the various mica-producing countries as sources of sheet mica, and often the figures may relate largely to scrap or inferior material, as is believed to be true of recent Russian figures.

The latest year for which fairly complete records of sheet production are available is 1934, and in that year the principal producing countries were then the following in order of importance: Russia, India, Madagascar, United States, Australia (doubtful as producing much sheet mica), Canada, Korea, Ceylon, Norway, Sweden, Brazil, Tanganyika, Bolivia, Southern Rhodesia and Northern Rhodesia. Figures for Argentina were not available, but that country is a fairly important source of sheet mica and should probably rank after Korea.

A digest of information relating to the occurrence and distribution of mica in the principal producing countries is contained in a comprehensive report dealing with all phases of the mica industry published in 1929 by the Canadian Department of Mines:<sup>24</sup> Mohr's book<sup>19</sup> is also a valuable work of reference on the subject.

#### POLITICAL AND COMMERCIAL CONTROL

The interdependence of the electrical and mica industries gives the mineral an economic and military importance not exceeded by any other raw material, and makes the matter of adequate supplies in wartime a subject of vital concern. During the World War, the ensuring of supplies of mica to the various Allied countries was one of the important activities of the British Imperial Ministry of Munitions.

India occupies such a dominant position as a producer of high-grade sheet mica as to make her the principal world factor in the supply of such material. The reserves are believed to be ample to take care of demand for an indefinite period. In addition, India enjoys a plentiful supply of cheap, native labor, enabling her to mine and prepare mica for market at costs greatly below those obtaining in most other countries. Since a large proportion of the electrical mica used is employed in the form of thin films or splittings for the manufacture of built-up mica plate, labor costs for this work, all of which is done by hand, are very important. Very

little mica is now split in countries outside of India; even Madagascar, as well as some other countries, ships part of its mica to India to be split.

Canada is one of the two important world sources of phlogopite mica, also used for electrical purposes, particularly where high heat resistance is required, and shares the world market for this class of mica about equally with Madagascar, a French possession. Other British possessions also contribute small and varying amounts of sheet muscovite to the total output, so that the British Empire may be said to have virtual control of mica supplies. It is doubtful whether any exigencies could bring about much important change in this situation, except at prohibitive cost.

While the United States is the world's largest consumer of mica, it produces normally only 15 to 35 per cent of its requirements of sheet mica larger than punch and circle sizes, and less than 5 per cent of its requirements of splittings; the output of punch and scrap mica, however, is sufficient to meet nearly all domestic requirements.<sup>18</sup> Domestic reserves doubtless are adequate to produce on an increased scale, but not in open competition, and only through the medium of subsidies and excessive tariffs could production be raised materially. In this connection it may be noted that during the war period the American production of mica remained practically constant, despite greatly increased prices and the urgent need for the material. The claim is sometimes made that American muscovite is inferior in its essential electrical and other physical properties to Indian mica: Horton,<sup>14</sup> however, disputes this, as a result of exhaustive tests conducted by the U. S. Bureau of Standards, and claims that any preference by consumers for the Indian product is due to the more satisfactory preparation and grading practiced by the Indian producers. A recent Indian official report\* estimates that of the combined sheet-mica production of India, the United States and Canada, India accounts for 87 per cent. American imports of electrical mica are drawn mostly from India, Madagascar, France and Canada, those from France presumably being re-exported material from Madagascar, since France herself produces no mica. By far the larger proportion of these imports represents mica splittings. Further small supplies are drawn from Great Britain (presumably re-exports from various Empire sources), Brazil, Argentina, British East Africa, Bolivia and Guatemala. Most of the South American product is trimmed sheet or slab mica. From these imports, plus the mica of home origin, are made practically all of the manufactured mica products required by domestic industry, together with a large surplus for export; imports of such mica manufactures are negligible. Mica enjoys a considerable measure of tariff protection, the duty in the United States being arranged on a variable scale, according to the class of product.

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\* Ref. 11, 37.



## USES

By far the greatest proportion of the sheet mica produced is employed for electrical insulation purposes, most of it being muscovite but a smaller amount of biotite being also employed. Biotite is softer and less heat-resistant. The larger sizes mostly go into heater elements for electric irons, toasters, etc., and into condensers, and the smaller sizes into segments, disks and washers, of which an almost infinite variety of shapes and sizes are made: these last are die-punched on power presses. Lamp sockets, fuses and radio-tube supports consume an important proportion of the punched washers and disks made. Some heater elements can use mica plate, compounded with special heat-resistant binders, and there is also a growing use for the same purpose of smaller sizes lapped and held together with eyelets. The greater part of the consumption of electrical mica, however, is used in the form of splittings, films around 1 mil thick and usually measuring 1 by 1 to 1 by 3 in. across; these are bonded together with shellac or a special synthetic resin (e.g., glyptal) and heat-pressed into plates of any required thickness. This plate can then be sawed, punched or molded into any sizes or shapes desired, and has insulating properties equal to straight sheet mica. Many types of mica plate are made for various special requirements, and splittings are also built up with paper or cloth to form flexible wrapped insulation. The laying and pasting of mica plate was formerly done by hand, but the work is now generally performed by mechanical layers.

Heavy-duty spark plugs, requiring high resistance to mechanical and thermal shock, as in airplanes, are built of mica washers bonded under pressure, the spindle being given a mica wrapping: only the best grades of phlogopite are used for the purpose.

Clear, transparent sheet muscovite is used for stove fronts, oven, furnace and kiln windows, lamp chimneys, canopies and shields; for the lenses of goggles, smoke helmets, gas masks, etc.; in projection apparatus, and for a wide variety of glazing purposes where resistance to breakage and heat is essential. Thin, translucent mica plate is sometimes used for ornamental purposes, chiefly in art lamp shades, shields and chimneys, and spotted muscovite is also used for similar purposes. While often found in large crystals, biotite is of little industrial use in the sheet form. Its high iron content gives it low dielectric strength, thus precluding its use for electrical purposes. Its black color and opacity, even in thin sheets, render it unsuitable for many of the purposes for which muscovite and phlogopite are used. Practically the only use to which it is put is in fine flake or powder form, as a coating or filler material, in the roofing, rubber, building, or other trades. Most of the biotite so used is obtained by pulverizing mica-rich biotite schist; some may also be recovered as a

by-product from the concentration of garnet, kyanite, feldspar and other minerals.

The better grades of wet-ground mica are employed principally as a decorative, luster-imparting ingredient in wallpaper, and also in some paints. It is used to some extent as a filler in rubber and other plastic compounds, but in general cannot compete in this field with other cheaper materials. Another use is for painting the water bags used in the vulcanizing of rubber tires, where it prevents sticking and gives the goods a fine finish. Wet-ground mica is of better quality than the dry-ground material, because it is usually prepared from a cleaner and better grade of scrap, and a better method of grinding and more care are employed in its manufacture.

A large proportion of the dry-ground mica manufactured goes to the roofing trade to be used as a backing for rolled asphalt roofing and shingles, to prevent sticking, and also to some extent as a decorative surfacing for these materials. Only the coarser grades are used for this purpose, but a proportion of finely ground mica is sometimes also incorporated into the body of the roofing itself. Relatively coarse mica is used to give a finish to stucco and concrete, and in the manufacture of artificial stone. Flake muscovite is used as Christmas-tree "snow," and for other decorative purposes: it is also employed, for its lubricating qualities, in mica axle grease, and as a dry lubricant. In the manufacture of tires and other rubber goods, mica powder is used as a dusting medium to prevent sticking in the molds, and it is used between the casing and inner tubes of tires to lessen friction and prevent sticking.

Ground mica, usually phlogopite, is used as an ingredient of certain types of molded electrical insulation, in which it is bonded with natural or synthetic resins, gums, asphalt, rubber, silicate cements or lead borate. Other miscellaneous minor uses include the annealing of steel, in explosives, in calico printing and sizing, and in lithographing.

Most of the expanded vermiculite sold is used for heat-insulation purposes<sup>22,25</sup> (see chapter XX).

#### PRODUCTION AND CONSUMPTION

Very complete figures on the mica industry in the United States are contained in the annual statistical reports of the Geological Survey and Bureau of Mines,<sup>18</sup> and these should be consulted for detailed particulars of production, imports, exports and consumption of the various mica products concerned.

The annual production of sheet mica in the United States in the 15-year period 1920 to 1934 has ranged from a peak of 2,172,159 lb., valued at \$400,184, in 1926, to a low of 338,997 lb., valued at \$45,882, in 1932. During the 11-year period 1920 to 1930, the annual production only once (in 1921) dropped below the million-pound mark, and in three

years exceeded two million pounds. A decided drop in production commenced in 1931, and persisted until 1934, when an upward swing was recorded. Consumption figures covering all classes of sheet mica are not given separately in the above reports, but consumption trends for the period 1923 to 1934 are indicated in Fig. 1.

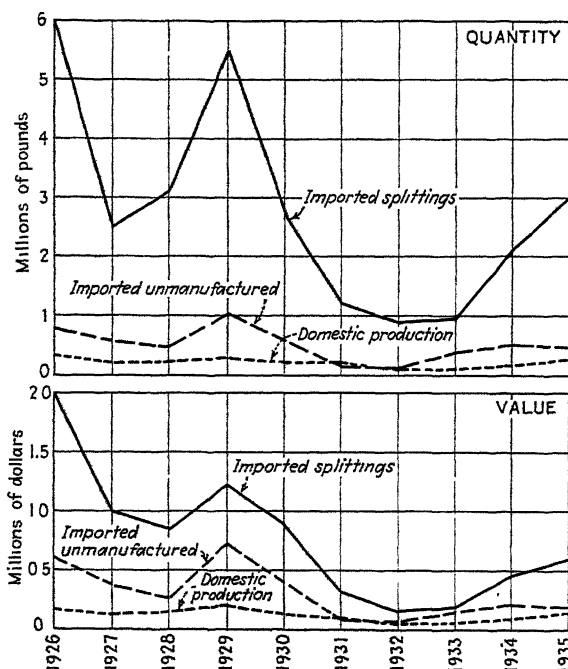


FIG. 1.—TRENDS IN IMPORTS OF SPLITTINGS AND OF BOTH IMPORTS AND DOMESTIC PRODUCTION OF THE BETTER GRADES OF SHEET MICA, 1926-1935, UNITED STATES.  
Minerals Yearbook, U. S. Bureau of Mines, 1936.

Scrap-mica statistics for the 15-year period 1920-1934 show annual production ranging from 2577 short tons, valued at \$56,849, in 1921, to 9695 tons, valued at \$173,537, in 1925: the average for the 10 years 1925-1934 was around 7000 tons. Imports of scrap mica have been recorded only since 1930, and show annual importations for the 5-year period to 1934 varying between 1360 short tons, valued at \$11,908, in 1932, and 3505 tons, valued at \$27,874, in 1934: most of the material comes from Canada, India, South Africa and Russia, in order of tonnage. All of this scrap is used in the production of ground mica, the demand for which has shown a steady increase.

The ground-mica trade is an important industry in the United States. Statistics are available only since 1923, and for the 12-year period 1923-1934 show annual productions ranging from 4866 short tons, valued at

\$304,660, in 1926, to 9831 tons, valued at \$398,681, in 1933. The production totals, however, also include a proportion of fine flake mica, recovered by washing kaolin residues and used without grinding in the manufacture of roofing materials. There has been a notable increase in the production of such washed mica in recent years. In 1934, thirteen companies were engaged in the production of ground mica, seven using dry-grinding processes and six grinding by wet methods: over half the plants are in the South, seven of them in North Carolina. The grade of product made, and its unit value, vary widely, depending on the nature of the raw material used, mine or shop scrap, washed flake, schist, etc., and on the method of grinding. The best grades are made from selected scrap muscovite by wet grinding and water-floating, and the lowest from dry-ground biotite schist. Small amounts of ground mica have also been imported during the last few years, mostly from Russia, the largest amount being 269 tons, in 1933.

No other country keeps as complete statistics regarding mica as the United States, and, as already indicated, world production, export and import figures often mean little or nothing because of lack of specification of the grade of product concerned. Only a few of the producing countries report their output other than as simply "mica." The data given here are taken from various sources, and mainly from the statistical reports noted in the bibliography at the end of the chapter.<sup>15-18</sup>

Ever since the early growth of demand for sheet mica to supply the electrical industries, India<sup>11,12</sup> has been the chief world source of the product. The industry has been said to employ at times over 15,000 hands, engaged in the various operations in and about the mines, trimming shops and splitting establishments. Export figures are usually taken as a truer index of production than actual production records, owing to the large amount of mica believed to be stolen from the mines and thus not reported. A further suggested cause of discrepancy between the two sets of records is that a large amount of small-sized mica, discarded years ago and relegated to the waste dumps, is now being reclaimed and made into splittings, such mica not being reported in production returns. However, export figures, on the other hand, may show an excess over actual production, because considerable amounts of foreign mica are shipped into India for conversion into splittings and re-exported in that form.

During the 15-year period 1920 to 1934, Indian sheet-mica exports have ranged from a low of 271 long tons in 1921 to a high of 1026 tons in 1934; for 11 out of the 15 years, the quantity exceeded 500 tons. Sheet-mica exports are greatly exceeded by exports of splittings, the amount of the latter having been from three to seven times that of the former during the same 15-year period. Actual tonnages ranged from a low of 1085 tons in 1921 to a peak of 4914 tons in 1929. The United States is

the largest purchaser of Indian mica, followed by Great Britain, France and Germany.

In recent years, India has become an exporter of scrap mica, and in 1934 the United States imported 1612 tons of scrap from India.

Canada,<sup>24</sup> once the principal world source of phlogopite mica, has registered a considerable decline in production of sheet mica in recent years, owing to increased competition from Madagascar. Canada produces little, if any, sheet muscovite. The phlogopite deposits are nearly all in adjacent sections of Quebec and Ontario, within a radius of about 100 miles of Ottawa. Hundreds of small pits have been opened up in this area at various times, but at present the entire production comes from only about half a dozen properties.

Detailed statistics on mica have been kept since 1921. During the 15-year period 1921 to 1935, production of sheet mica fluctuated widely, from a peak of 1,198,004 lb. in 1924 to an extreme low of only 2019 lb. in 1932. A pronounced decline began in 1928, when production dropped to 91,064 lb., from nearly 700,000 lb. in the preceding year. Production of splittings has followed a closely parallel course, dropping from a high of 210,056 lb. in 1923 to a low of 3350 lb. in 1932.

Canada produces considerable scrap mica, most of it recovered from old mine and trimming-shop dumps. Scrap output has ranged from 306 tons in 1932 to a high of 3993 tons in 1929. Most of the material is shipped to grinding mills in the United States; the domestic grinding industry is small, and consumes only a few tons annually.

Madagascar<sup>10</sup> produces both muscovite and phlogopite mica, but the proportion of the former is comparatively small. The Madagascar mica industry dates from 1920, with initial exports in that year of 34 tons. In the 15-year period to 1934, exports have risen to a peak of 635 tons in 1928. The proportion of splittings included in the export totals is not stated, but most of the product is believed to be sheet or block mica. Part of the Madagascar splittings offered are understood to be made in India. While the average run of Madagascar phlogopite is probably not superior in actual quality to the Canadian product, the method of trimming and grading has been very much better; only perfect, sound sheets, sickle-trimmed to remove all waste, being offered. As a result, and with the advantage of much lower labor costs for mining, trimming and splitting, Madagascar mica has secured a firm hold in the world market, with consequent detriment to the Canadian industry. Canada even imports a certain amount of Madagascar splittings to supply her own requirements. Most of the Madagascar mica exports go to France, the United States and Great Britain, in order of tonnage.

In South America, Argentina and Brazil both produce muscovite, but at a fluctuating rate. Some of it is extremely good. A considerable proportion of the output goes to the United States. Brazilian mica

exports have averaged around 50 tons for the last 15 years, and Argentina exports, 80 tons; no particulars of grade of product are available, but the figures are believed to refer mainly to trimmed sheet. Guatemala has also supplied a small quantity of sheet muscovite from time to time.

In Asia, Korea produces small amounts of both muscovite and phlogopite. Since 1923, statistics show an average annual output of about 20 tons, with no grade of product specified. In 1934, production rose to 101 tons. Ceylon has a small and intermittent production of phlogopite, amounting to a couple of tons a year: in 1934, however, the exports showed a sharp rise to 20 tons. Cochin China is also stated to produce a small amount of phlogopite, but no output for this country is shown in any of the statistical tables covering world mica production. Japan is credited with a small mica industry, but the material is believed to be mostly fine flake, or scrap, which is utilized for grinding for various purposes. It is possible that some of the recorded Russian production of mica is derived from deposits in the Asiatic U. S. S. R., but no data on this point are available.

In Africa, small amounts of sheet muscovite have been produced intermittently in Kenya, Nyasaland, Cape Province, Natal, the Transvaal and Northern Rhodesia. The most important continental African producers, however, are Southern Rhodesia and Tanganyika. Between 1921 and 1931, the Southern Rhodesian output (all muscovite, no grade specified) ranged from 66 to 183 tons. There was a sharp falling off in 1931, and the production has since declined steadily to only 2 tons in 1934. Production of mica (muscovite) in Tanganyika (formerly German East Africa) commenced under German rule, and after the war was continued under British Government control. Since 1921, the annual output has ranged from 3 to 68 tons, with no grade specified. Production has fallen off materially since 1930, with an average for 1931 to 1934 of only 10 tons. Between 1924 and 1931, the Transvaal recorded a considerable annual production of scrap muscovite, with amounts ranging from 843 to 3572 tons: a considerable proportion of this scrap is believed to have been exported to the United States for grinding. Total scrap exports in 1928 amounted to 3245 tons. A small mica-manufacturing industry exists in the Transvaal, with plant at Johannesburg.

Australian mica production has shown a considerable and steady increase since 1924, output having risen from 2 tons in that year to 138 tons in 1934; no particulars of the grade represented are given. Most of the material is believed to come from deposits in south, central and western Australia. In the last named, an unusual quality of lepidolite is found, in large, clear sheets. This was formerly mined on a small scale for gramophone diaphragms, on account of its high resonant property.

With the exception of the United States, Canada, and, perhaps, Russia, none of the more important mica-producing countries consume

any large share of their sheet-mica output, the great bulk of it being exported to the principal countries that manufacture electrical equipment of which the principal ones are the United States, Great Britain, Germany, France and Italy. The United States appears to be the only country for which detailed statistics of mica consumption are available, therefore it is not possible to give any very precise indication as to the general world consumption by users. Mica imports are often a misleading index of consumption, since in countries like England and France a proportion of the mica imported from India and Madagascar is re-exported to other countries. Canada, for instance, imports considerable mica from the United States, and smaller amounts also from Great Britain and France, in addition to the supplies drawn direct from India, all or most of such mica probably being of Indian or Madagascar origin.

A report<sup>15</sup> dealing in a comprehensive way with the world trade in mica was issued a few years ago by the U. S. Department of Commerce, and this should be consulted for nearer details on the subject.

#### PROSPECTING AND EXPLORATION

Prospecting and exploration methods for mica call for little comment. Granite pegmatite dikes, to which the occurrence of sheet muscovite is confined, are sufficiently conspicuous rock masses readily to attract attention within any area intruded by them, and mica is a mineral extremely resistant to weathering influences, so that the crystals or books remain fresh and unaltered under almost any conditions. Even kaolinization, which, in extreme cases, may reduce all or most of the feldspar in a pegmatite to clay, effects no change in any mica present, and considerable muscovite is recovered from more or less heavily kaolinized dikes, particularly in tropical countries, where the zone of surface weathering often penetrates to considerable depths.

The phlogopite-bearing pyroxenites, also, while perhaps not as conspicuous rocks as the granite pegmatites, and often less clearly demarcated in their boundaries than the latter, are yet fairly readily distinguishable in the field, and, once found, can be easily followed when not obscured by overburden.

While, in both types of rock, the mica crystals may be irregularly distributed through a dike mass, often they tend to follow more or less definite zones. Muscovite commonly occurs either in localized shoots, which may lie in any position within a dike, or is confined principally to one or other of the contact zones. Phlogopite, on the other hand, more generally occurs as rich concentrations in more or less veinlike bodies, or in irregular pocketty masses of calcite within a pyroxenite mass.

Muscovite deposits, in general, are more persistent than phlogopite deposits, and therefore can usually be more readily followed to depth.

Phlogopite deposits are inclined to be decidedly "spotty," and the richer zones or pockets often peter out to narrow unprofitable stringers without warning. Such stringers, if followed, may again widen out into commercial bodies, but the amount of dead work involved in following them is often discouraging and frequently causes abandonment of pits at a comparatively shallow depth. Even old and experienced phlogopite miners concede that they have no useful indications to guide them in following mica shoots, and maintain that the mineral, more perhaps than any other, is "where you find it."

Diamond drilling for mica is of limited usefulness, owing to the disseminated or localized form in which the crystals occur, and is seldom used. The usual practice is to follow a shoot or zone as long as it is profitable to work it and then to prospect by surface trenching for other bodies.

#### MINING METHODS

Methods of mica mining<sup>19</sup> are usually of the most elementary nature, and call for only brief description. Outside of India, most of the mica produced is won from open-cast quarries, many of them comparatively shallow surface pits, and few mica mines have been opened and developed by orthodox underground mining methods. Only rarely are mica-mining operations undertaken with the investment of an important amount of capital for plant, and mining usually proceeds in a fortuitously progressive, and often intermittent, manner, depending on the success attending initial surface operations in finding of bodies rich enough to follow to depth.

Blocking out of mica-bearing ground ahead of production is usually impracticable, and reserves are a matter of chance except in unusually rich and well defined deposits. When mining is carried much below surface, the workings seldom are planned but simply follow the shoots or individual crystals or bunches of crystals. This often results in a series of most irregular drifts and stopes. In exceptionally wide pegmatites, such workings are sometimes later glory-holed, subsequent mining being conducted by open-pit methods. As a rule, however, hoisting equipment at mica mines is kept to a minimum, and no more ground is broken than is absolutely necessary.

On level or sloping ground, a series of small pits is often opened along the strike of a pegmatite outcrop, and these may be joined later into a single large open cut. Where a vertical dike outcrops in the side of a steep ridge, mining is sometimes conducted from one or more adits driven in along the strike at different elevations, and the mica-bearing ground between taken down by stoping methods. In flatly dipping pegmatites (sills), the richer concentrations of mica are not uncommonly found to



follow one or other of the contact zones, and the usual practice is to put down inclined drifts following the dip, which later may be joined by lateral galleries.

Little timbering is required in the average mica-mining operation except in lagging the collars of hoist openings. For shallow workings, derrick hoists are commonly employed, replaced by drum hoists when depth is reached. Where wood fuel is plentiful, steam power is still sometimes used, but availability of the light, portable, gasoline-driven compressor has greatly reduced the number of mines using steam-driven engines and drills. In regions where electric power is available, large established mines sometimes use electric equipment, but, outside of India, the number of mica mines in consistently steady production is limited, and for intermittent operation the cost of current is prohibitive.

The value of mica being essentially dependent on its physical perfection, care must be used in blasting, to ensure the least possible damage to the crystals, the object being to break the rock without shattering it. The same care is required in mucking. The mica is carefully sorted out from the broken rock in the pit and placed in boxes for conveyance to the cobbing benches; here, adhering rock is removed and the crystals are broken into slabs or sections thin enough to be handled by the trimmers.

A report of the U. S. Bureau of Mines,<sup>27</sup> published a few years ago, gives an analysis of costs and recoveries at two muscovite mines in North Carolina. In one of the mines, the value of the rock broken, in block mica per ton, was found to be \$3.46; with scrap mica and merchantable feldspar added, \$3.93. In the other mine, where no scrap or feldspar was produced, the recoverable value was \$2.55 per ton. These values show that even when a pegmatite is considered rich enough to be worked, the recoverable mica per ton of rock broken is low. Against these values, it was found that mining and trimming costs totaled \$2.75 and \$2.23 per ton of rock, respectively.

The merchantable value of mica rises rapidly with increase in the size of the trimmed sheet yielded by crystals, and the profitable operation of a mine is therefore essentially dependent on the average size of the mica crystals. This is a highly variable factor, and at the two mines included in the analysis above, the proportion of sheets grading 3 by 5 in. and up in one was 24 per cent of the total mica produced and in the other was less than 1 per cent.

#### PREPARATION FOR MARKET

##### SHEET MICA

The rough mica crystals, or "books," taken from the mine are first of all subjected to a preliminary cobbing operation, whereby attached rock is hammered off and the crystals are broken into slabs or sections

of convenient weight and thickness to be handled by the trimmers; a first, preliminary separation is also made of merchantable sheet from waste scrap material, the latter being sent to the scrap-storage pile for shipment to a grinding plant. In remote localities, such scrap may not be worth its transport, and goes to the rock dump. At small mines, and depending on prevailing climatic conditions, cobbing may take place in the open or, more usually, in sheds furnished with benches at which the workers sit and to which the mica falls through openings connected with storage bins.

At small mines, rough-cobbing may be the only stage of operation practiced in the conversion of run-of-mine product to merchantable mica, the material being sold in that form to dealers that carry out the other operations at centrally located establishments (so-called mica "shops"). The preparation of mica is a skilled operation, calling for experience in gauging quality, as determined by color, inclusions, splittability and freedom from cracks, flaws and crystallographic imperfections, and in determining the maximum surface area that can be obtained from a given sheet by trimming; considerable manual dexterity is required, also, in the actual splitting and trimming operations themselves. Unskilled labor may easily cause avoidable waste and unsatisfactory trimming and grading, and it often pays the smaller mine operator better to leave these operations to regular dealers that employ labor qualified for the work.

Mica is prone to structural imperfections, and often deposits are found in which the large bulk of the crystals present is practically worthless as sheet mica. The terms "tangle-sheet," "herringbone," "A-mica," etc., are applied to mica whose quality is seriously impaired by crystallographic disturbance. "Wedge" crystals are crystals that are thicker on one side than on the other, yielding sheets of unequal thickness. "Ruled" mica indicates sheets that have developed parallel fracturing, resulting in their breaking into narrow strips when split. Distortion of mica crystals, due to squeezing or crushing caused by rock movement, is also common, and results in creased or wavy sheets. In addition, the outer portions of mica crystals are often hard and brittle, owing to the presence of intergrown foreign mineral substance, and the sheets exhibit cracks and flaws around their edges. It is readily apparent that the securing of sound, perfect mica, especially of the larger sizes, free from all flaws and imperfections, is attended by a large amount of waste, and, indeed, the percentage of merchantable sheet mica yielded by a deposit is often only a small fraction of the run-of-mine production.

After being cobbled, the mica is ready for rough-splitting and trimming. The first-named operation, in the United States usually termed "rifting," splits the rough slabs to a thickness of  $\frac{1}{16}$  in. or less, a thickness at which the mica can be readily cut by a knife. The rifting operation is a manual one, the operator first using a light hammer to loosen the laminae on one

side of a crystal and then inserting between them the point of a stout, short, two-edged mica knife, specially made for the purpose. In this operation, a further discarding of unsound mica takes place, any crushed or buckled material and portion of crystals that will not split owing to crystal imperfections or to the presence of included foreign mineral substance being relegated to the scrap dump.

After this preliminary rough-splitting, or rifting, the sheets pass to the trimming shop, where other workers remove all edge waste and any unsound mica within the sheets. The smaller sizes, known as punch or washer stock and used for the die-punching of small washers, disks and segments, are often left untrimmed. Trimming may be rough or close, depending on the care given to the operation. Formerly, much of the Canadian production was marketed in "thumb-trimmed" form, whereby only the ragged edges of the sheets were roughly broken off by hand, with a minimum use of a knife. Such mica usually contained a large proportion of unsound waste, and the practice has now largely been discarded in favor of closer knife-trimming. Trimmed sheet mica has irregular outline, the size and shape of the sheets being merely the result of the waste removed, and is generally termed "block," "uncut," or "unmanufactured" mica, to distinguish it from "slab" mica, which indicates sheets cut to specified, rectangular dimensions.

The trimming operation is variously performed in different countries. It may be done with a small hand knife, which is used to give a bevel or bias slice, the resulting bevel edge aiding in further splitting of the trimmed sheets. In the Bihar field, in India, and in some other countries, this operation is performed with a sickle, and the mica so trimmed is known as "sickle-trimmed." In the Madras field, in India, trimming is done with a pair of shears and the product has straight, unbeveled edges. All of these methods of trimming are essentially hand operations. Mica, usually the smaller sizes, may also be machine-trimmed (sometimes also termed "knife-trimmed"), and for this, a power-driven, vertical wheel or disk is employed, to one side of which two pieces of steel are bolted, to act as knives. The wheel revolves in a slotted bedplate, and the mica sheets are held by hand on the plate and pressed against the wheel, the steel lugs shearing off the edges.

In trimming mica, a wide range of sizes of sheet is produced. For marketing, the sheets are graded both for size and for quality. The size classification varies in different countries, there being no uniformity. Probably no other mineral product is as difficult to classify as mica or is so variable in quality on account of the wide range of sizes and grades produced.

In the United States, sheet muscovite is graded into three principal classes according to quality: (1) clear, (2) slightly stained or spotted, and (3) heavily stained or spotted. The size classification is: "punch,"

yielding a circle  $1\frac{1}{4}$  to  $1\frac{1}{2}$  in. in diameter; "circle," yielding disks up to 2 in. in diameter, followed by a series of dimensional grades ranging from  $1\frac{1}{2}$  by 2 in. to 8 by 10 in. and over. In Canada, phlogopite mica sizes are also designated dimensionally, from 1 by 1 in. upwards; Madagascar follows the Indian system of arbitrary numbers. Indian mica, which represents by far the largest amount used, is graded for size by a series of numbers from No. 6 (the smallest) to No. A-1 (the largest), sizing being done by laying the sheet on a ruled chart or templet to determine the area. Above the A-1 grade, there are three larger special sizes, designated by dimensions up to 80 sq. in. or over. Indian grading for quality embraces nine classes, from clear to badly stained, so that there are over 60 combinations of size-quality grades into which Indian mica may fall. Phlogopite mica (which is never spotted or stained as muscovite is) is graded for quality by its color, such as "light silver amber," "dark silver amber," "wine amber," etc., the lighter shades being the softer and better splitters.

The larger sizes of trimmed mica are marketed for various uses in the sheet or block form, but the smaller sizes, which represent by far the larger part of the production, are utilized mainly for the making of splittings. Splittings are made by hand. Numerous attempts have been made to devise equipment for splitting mica mechanically, and a wide variety of devices have been tried out. One recent method employs heating of the sheets, followed by cooling in water, drying, and carrying the sheets between a pair of belts that pass over and under a series of rods, thus bending the sheets and causing the laminae to slip over one another and separate. Fair success is stated to have been attained by this process in commercial practice, working on partly weathered phlogopite recovered from old waste dumps.

### GROUND MICA

Considerable tonnages of waste scrap mica are now ground to produce mica powder for a number of industrial purposes. The industry relies largely for its raw material on mine and trimming-shop scrap muscovite purchased from the sheet-mica producers. Schist flake mica is also used extensively for the same purpose, the principal deposits of such mica schist, both muscovite and biotite, being in North Carolina. There is also a growing production in that state of flake muscovite recovered by the washing of kaolin residues from mines in the Spruce Pine district.

The type and degree of preparation varies with the use to which the material is to be put, and both wet grinding and dry grinding are employed. For certain purposes, it is desirable to retain as far as possible the natural slip and luster of the flakes, and to this end the material is ground wet. Wet grinding is usually done in chaser mills—circular tanks

about 10 ft. in diameter and 40 in. deep, in which large wooden rollers rotating on horizontal arms revolve about a central shaft. The tank bottoms and the rollers are made of end-grain blocks of hardwood, and the diameter of the rollers is 30 to 40 in., with faces of 20 to 24 in. Complete grinding of a 1-ton charge of mica takes 4 to 8 hr., depending on the nature of the feed and the speed of the mill. Water is added at intervals during the operation, so as to keep the charge in a pasty condition, and the friction generated is sufficient to raise the temperature to boiling point. Careful control is required in order to ensure maximum grinding efficiency without overheating of the charge and consequent damage to the mica. When grinding is complete, the contents of the tank are sluiced out into launders and sand boxes, where the gritty impurities and coarse mica settle out, the fine mica being carried over to settling vats. After the excess water is siphoned off, the mica sludge is dried on steam tables, either with or without previous filter-pressing, run over scalpers to remove any coarse particles and finally bolted to size. Products as fine as 350 mesh are obtained by the process; the coarser oversize is returned for regrinding. Other methods of wet grinding have been employed to a limited extent, including grinding in ball mills, but such processes are not commonly used.

In the dry grinding of mica, machines of the high-speed, hammer-mill type, equipped with air separators or screens, are usually employed. For effective grinding, the feed requires to be dry, and it may be necessary to first pass it through a drier, generally one of rotary type. Mica, while soft, is tough and resilient, and the flakes have to be reduced by tearing or shredding, rather than by impact. Screen pulverizers are generally preferred, the discharge through the screens being then bolted to the required mesh sizes. Dry-ground mica, particularly the finer sizes, is usually of not nearly as high quality as the wet-ground product, and often includes a considerable amount of gritty impurities derived from rock matter contained in the scrap used. Some of the dry mills in the United States use mica schist as raw material for grinding, and chlorite schist is similarly sized used.

A proportion of the North Carolina production of mica powder consists of fine flake mica recovered by washing methods from kaolin residues, the material being sized by screening and sold without grinding.

#### TESTS AND SPECIFICATIONS

Sheet mica is purchased in the first instance on visual inspection, and dealers subject samples to close examination for physical imperfections, such as cracks, pinholes, degree of spotting, foreign mineral inclusions, splitting quality, etc. Relative hardness and color are also important factors in gauging quality, and where dealers draw supplies from a

number of sources, care is taken to ensure uniformity in these respects in making up shipments from stock. Phlogopite mica is variable in all the properties that affect its industrial value, and different mines in the same area often yield mica of widely differing quality and value; accordingly, care is taken not to mix shipments from different deposits. Miners and dealers seldom have the facilities for conducting any heat or electrical tests on mica, and such tests are usually made by the larger manufacturing firms that purchase the material, or the tests may be made for industrial concerns by scientific institutions having the necessary equipment for the purpose.

Specifications called for in mica naturally vary with the use to which it is to be put. Stove mica is required to be transparent, free from spots or blotches, and capable of enduring high temperatures without change. For electrical mica, dielectric strength, power factor, heat resistance, softness and flexibility are all important considerations, but the rigidity of the specifications varies with the particular use it is required to serve. For general electrical purposes, however, all physically sound muscovite and phlogopite possesses sufficiently high dielectric strength to obviate risk of puncture. Phlogopite is softer than muscovite and formerly was specified for use in commutator segments, as it reduced wear, but the practice of undercutting enables muscovite to be used for the same purpose. For condenser purposes, mica is required to have a low power factor, in order to obviate heating under load—only the best grades of muscovite are used in condensers, and the films are subjected to rigid X-ray inspection to detect flaws and inclusions. Heat resistance is important for mica to be used in electrical heater equipment, such as irons, toasters, etc., and phlogopite, which will withstand temperatures up to 1000° C., is generally used. Some phlogopites, however, bloat when heated, because of incipient hydration, and mica required for heater-plate use requires testing for heat resistance before use. For mica used in electrical wrappings, flexibility is important, and the softer phlogopites are generally specified for this purpose. One of the most exacting uses for which mica is employed is in heavy-duty spark plugs, and only sound phlogopite possessing to a high degree all of the properties mentioned above is suitable.

Detailed descriptions of the methods employed by the U. S. Bureau of Standards in the testing of mica for its dielectric strength and heat resistance, are given by Horton.<sup>14</sup> Tentative methods of test for grading sheet mica, and for testing mica plate built up from splittings, are described in Tentative Standards, 1934, of the American Society for Testing Materials, pages 836-842.

For most of the uses for which ground mica is employed, no very exacting specifications exist. For the better grades of wet-ground mica, used principally in the wallpaper trade, good white color and high luster

are requisite, as well as freedom from gritty impurities: the flake sizes used range to as fine as 325 mesh. It is naturally desirable for general uses that the ground product should not contain an undue amount of foreign mineral matter, but considerable latitude in this respect is often allowed for the lower grades, where cheapness is a consideration. Color is seldom of much importance, and all shades from black to white are acceptable for most purposes. Flake particles having more or less equidimensional form, as opposed to fibers or shreds, are desired for the coarser meshes, where covering power is a consideration, as in the roofing trade. The particle size demanded varies widely with the use, from 10 to 60 mesh for roofing purposes to minus 200 mesh for the rubber trade and for various filler uses.

#### MARKETING

The marketing of sheet mica is often an involved process, and the material frequently passes through a number of hands before reaching the consumer, because of the many small mines contributing to the supply, the operators of which are not familiar with trade requirements and lack the capital to do much more than take the mica out of the ground and roughly cob or trim it. They sell it in this form to local jobbers or dealers. The latter often buy the output from a number of mines, and run a central mica shop, employing the necessary skilled labor to trim and grade the rough sheets, reselling the product to larger dealers who, in turn, resell to mica brokers or consign, on order, lots of specified sizes to the actual consumers. There is probably no other mineral product that passes through as many channels between mine and user as does mica. The small producer is often inclined to regard the middleman in the industry as unnecessary evil, who takes an undue share of the profits, but the actual miner seldom has the necessary capital to maintain adequate stocks from which he can fill orders for any specified grade or size at short notice, and he may not have the requisite knowledge of trade standards and specifications or of demand and price trends, so that the dealer forms a useful link between miner and ultimate consumer. The larger, steady producer is in different case, and often ships direct to consumers familiar with the quality of his mica, thus netting a greater share of the profit. Ground mica, being a bulk commodity and prepared in finished form at the grinding mill, is not usually bought for resale but is shipped direct to the consumer.

#### PRICES

Mica prices are difficult to review briefly. The subject is complicated by the large variety of grades offered, by the passage of the product through successive hands, and by a system of trade discounts. There are virtually no standard quotations, and dealers are often reticent on

prices, owing to the intensive competition.<sup>1</sup> Purchases are usually made on individual contract by the larger consumers, through accredited agents; by dealers, on the basis of samples submitted by producers. London is the principal clearing center for much of the world's mica, a large proportion of the Indian production being shipped there for sale and re-export. Much of the Madagascar output is sold through French houses. Formerly, fortnightly mica auctions were held in London, which were attended by consumers or their agents, who bid ex-warehouse for their requirements, but this system was abandoned after the war.

Production and trade statistics are of little value as an index of prices, owing to the obvious difficulty of separately listing all of the many grades dealt in. Very few countries classify their mica imports and exports, all mica usually being lumped together regardless of its character, and the totals may include high-grade sheet, splittings, scrap, and even also ground mica, the values of which range between several dollars and a

TABLE 1.—*Prices of Uncut Sheet Mica in 1935<sup>a</sup>*  
Per Pound, United States

Size, In.	Price	Size, In.	Price
Punch . . . . .	\$0.03–\$0.05	3 by 4 . . . . .	\$0.90–\$1.25
1½ by 2 . . . . .	0.15–0.30	3 by 5 . . . . .	1.25–1.50
2 by 2 . . . . .	0.30–0.50	4 by 6 . . . . .	1.50–2.50
2 by 3 . . . . .	0.50–0.65	6 by 8 . . . . .	2.50–3.50
3 by 3 . . . . .	0.65–0.85	8 by 10 . . . . .	4.00–5.00

<sup>a</sup> From Minerals Yearbook, 1936, U. S. Bureau of Mines.

TABLE 2.—*Approximate Average Prices of Uncut Sheet Mica in New York in 1935<sup>a</sup>*  
Per Pound

Domestic			India (duty paid) <sup>b</sup>					
Size, In.	Clear	Black Spotted	Size No.	Clear and Slightly Stained	Fair Stained	Good Stained	Stained	Black Spotted
1½ by 2 . . . . .	\$0.40	\$0.15	6 . . . . .	\$0.94	\$0.64	\$0.34		
2 by 2 . . . . .	0.60	0.25	5 . . . . .	2.14	1.69	1.46	\$0.54	\$0.33
2 by 3 . . . . .	0.90	0.40	4 . . . . .	3.04	1.84	1.69	1.09	0.64
3 by 3 . . . . .	1.20	0.55	3 . . . . .	3.35	2.59	2.14	1.69	0.99
3 by 4 . . . . .	1.40	0.75	2 . . . . .	3.79	3.04	2.74	2.29	1.47
3 by 5 . . . . .	1.60	0.90	1 . . . . .	4.54	4.09	3.64	3.19	1.99
4 by 6 . . . . .	1.90	1.00	A-1 . . . . .	9.04	6.04	5.59	4.54	2.14
6 by 8 . . . . .	2.40	1.10	Special . . . . .	12.04	8.44	6.04	5.44	2.22

<sup>a</sup> From Minerals Yearbook, 1936, U. S. Bureau of Mines.

<sup>b</sup> Prices of India mica calculated on basis of £1 equals \$4.80.



fraction of a cent per pound. Since most of the sheet mica used in the United States is imported, prices are subject to considerable and frequent variation caused by fluctuations in the currencies of the various producing and re-exporting countries. Such fluctuations often also cause periodic diversion of shipments from one market to another.

In view of the foregoing, prices can conveniently be shown only in terms of the latest available figures relating to the domestic market. The prices shown in Table 1 serve to indicate the rapid appreciation in value with increase in sheet size; also the wide variation in value due to quality.

From information furnished by New York importers, sheet mica prices generally in 1935-1936 stiffened slightly over the 1934 figures shown in Tables 1 and 2, but with notable increases of from 30 to 45 per cent for the largest and best grades of Indian muscovite. The price of both Indian and Madagascar splittings remained substantially unchanged. Dealers report 1935 prices for Madagascar sheet phlogopite, of hard, heat-resistant type, as given in Table 3.

TABLE 3.—*Average Prices of Madagascar Uncut Sheet Phlogopite*  
Per Pound, ex Dock and Duty Paid at New York, 1935

No.	Size <sup>a</sup> , Sq. In.	Price	No.	Size <sup>a</sup> , Sq. In.	Price
5	3 to 6	\$0.82	1	24 to 36	\$3.45
4	6 to 10	1.75	A-1	36 to 48	3.99
3	10 to 15	2.09	00	48 to 60	4.40
2	15 to 24	2.64	000	60 to 80	4.75

<sup>a</sup> Approximate size of rectangle.

Canadian producers report 1936 prices of first-grade, silver-amber phlogopite as shown in Table 4. Prices of Indian and Madagascar splittings are given in Tables 5 and 6.

TABLE 4.—*Average Prices of Canadian Phlogopite*  
Per Pound, F.o.b. Ottawa, 1936

Knife-trimmed Sheet		Splittings	
Size, In.	Price	Size, In.	Price
1 by 1....	\$0.20	1 by 1.	\$0.48
1 by 2....	0.30	1 by 2.	0.50
1 by 3....	0.40	1 by 3.	0.55
2 by 3....	0.60		
2 by 4....	0.90		
3 by 5..	1.50		
4 by 6..	2.25		
5 by 8..	3.00		

TABLE 5.—*Average Prices of Indian Muscovite Splittings*  
Per Pound, ex Dock and Duty Paid at New York, 1934

Grade	Price	Grade	Price
No. 4: book-packed <sup>a</sup>	\$0 93	No. 6: book-packed . . . .	\$0.50
Loose . . . . .	0.75	First-quality loose . . . . .	0.23
No. 5: book-packed . . . .	0.83	Second-quality, loose . . . .	0.17
Loose . . . . .	0.45	Third-quality, loose . . . .	0.13
No. 5½: book-packed . . . .	0 75	Cheap: loose . . . . .	0.10
Loose . . . . .	0.30		

<sup>a</sup> "Book-packed" refers to the system used in making splittings in India, whereby all the films split from a single block or sheet are laid together, like a deck of cards, the films being first dusted with powdered mica to keep them from sticking together. In this form, the splittings are ideal for laying in the making of mica plate.

TABLE 6.—*Average Prices of Madagascar Amber Mica Splittings*  
Per Pound, ex Dock and Duty Paid at New York, 1934

No.	Size, Sq. In.	Price
4	6 to 10	\$0 65
5	3 to 6	0 45
6	1 to 3	0 40

The average prices of splittings consumed in the United States in 1934 were: Indian muscovite, \$0.25; Canadian amber, \$0.40; and Madagascar amber, \$0.42. Prices of Indian, high-grade, fair-stained condenser mica, in the form of splittings 1 to 3 in. thick, ex dock and duty paid, at New York, in 1935, were as follows: No. 5, \$2; No. 5½, \$1.45; No. 6, \$1.20.

The price of scrap mica varies with the quality, whether mine-run, dump waste or selected shop scrap. The average price paid for domestic scrap in 1935 was \$12.94 per short ton, and \$12.93 in 1934. Canadian scrap phlogopite is currently quoted (1936) at \$9 per short ton, f.o.b.

The average price of domestic, dry-ground mica in 1935 was \$22.52 per short ton. The price of wet-ground mica varies widely, according to grade, the average price in 1935 being \$63.96 per short ton, compared with \$90.81 in 1934. Canadian dry-ground phlogopite is currently (1936) quoted as follows, all prices per short ton, f.o.b. Ottawa: minus 20 plus 60-mesh, \$25; minus 60 plus 120-mesh, \$30; minus 120 mesh, \$45.

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## CHAPTER XXVII

### MINERAL FILLERS

BY ALDEN H. EMERY,\* MEMBER A.I.M.E.

**F**INELY ground materials, usually of mineral origin, are added to asphalt, paint, paper, rubber and many other products. Although sometimes these mineral fillers are solely cheap, inert diluents, often they are added to improve the properties of the finished product. Most people think of them as minor constituents; others call the whiting in putty a filler, although putty is composed only of linseed oil and whiting. An exact definition, then, to cover all modern uses is extremely difficult. Perhaps the one to which there can be the least exception is: Mineral fillers are finely ground materials of mineral origin used, for other than pigment purposes, in compounding various products and which, although sometimes actively changing the properties of the product, are themselves unchanged in compounding or use. Fertilizer fillers are materials that contribute none of the three major plant-food elements in an available form.

#### MATERIALS USED AS FILLERS

The author of this chapter has often prepared lists of mineral fillers. It is a waste of time and space. Any finely ground material can be used somewhere, many interchangeably, although suitable fillers for any one product may be limited. With substitution so easy, the field is highly competitive; in the final analysis economic conditions usually are the controlling factors. Thus, a fine by-product that otherwise would be a waste material has a distinct advantage over something that must be ground to order. Fine grinding is expensive; yet rock flour is made in the crushing, cutting or shaping of many rock products. Fine precipitates are the by-products of some chemical processes; frequently they present a serious disposal problem. These fine wastes are the logical mineral fillers, since presumably they can be obtained very cheaply.

No product is marketed with less scientific exactness than mineral filler. An industry or a company finds one material that gives satisfactory results and thereafter always uses that filler. As a result, the demand may increase until the waste or by-product originally offered is no longer sufficient and the filler must be ground or manufactured especially to meet the demand. So great are the prejudices of individual

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users in favor of such selected products that they are willing to accept the necessary increase in price rather than risk a change to another material. There is some justification for this, because change in filler might necessitate change in batch formulas or methods of handling that would lead to disruption of the manufacturing process, even if only temporary, and might be more costly than the adherence to a single proved filler. Nevertheless, it does mean that the producer of *assured large quantities* of a very cheap waste suitable for filler might make inroads on an established product.

Suitable materials include pulverized minerals and rocks, such as clay, limestone and talc; naturally occurring particulate matter, such as sand; materials prepared directly from minerals and rocks, such as lime and cement; chemically prepared fillers, such as *blanc fixe*, precipitated whiting and zinc oxide; and various forms of carbon black, indispensable to the rubber industry.

#### FILLER-CONSUMING INDUSTRIES

The use of mineral fillers is widespread, but because of the lack of published statistics on their consumption\* it is impossible to say with certainty where the greatest tonnages are used. Some of the more important consuming industries are asphalt, fertilizer, molded products, paint, paper and rubber. Fillers also are used in cosmetics, dynamite, insecticides (as carriers), linoleum, lubricants, mortars, oilcloth, polishes, putty, stock feed (as a source of certain chemical elements), wallpaper, et cetera.

*Asphalt.*—In asphalt paving mixtures the filler is added to reduce voids and to increase the resistance of the pavement to displacement under traffic. The effect of several variables on these factors was discussed in detail by Emery in 1933;<sup>7</sup> subsequent research has not changed the picture there presented. In general, it can be concluded that most inert mineral matter may be used successfully as a filler for sheet-asphalt paving mixtures if the material is ground fine enough, if it has an even grading of sizing, and if the mixture is scientifically designed. For many years the industry took the position that only limestone dust and Portland cement made satisfactory fillers; this is not true. There may be more difference between results using two samples of limestone, or two samples of any other material, than between two different materials, provided the proper mixture for each filler is used. What constitutes a satisfactory formula using one filler is not necessarily suitable with another. The proper mixture can be determined only by laboratory test, which should be carried out on all proposed filler prior to its use.

Fillers also are used in bituminous cements, asphalt tile, battery boxes, mastic and bituminous expansion joints for concrete pavement. In the

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\* The fertilizer industry is the only exception.

<sup>7</sup> References are at the end of the chapter.

latter material, 25 to 50 per cent of filler may be used. It increases the toughness and resistance of the mixture to temperature change. For battery boxes, the filler must be resistant to the electrolyte.

*Bituminous Sheet Roofing.*—In making sheet roofing, a fabric is saturated or coated or both saturated and coated with a bituminous substance. Pulverized mineral matter may be used either as a filler in the asphaltic mixture or as a surfacer. Abraham<sup>1</sup> gives three reasons for mixing filler in the surface coating: (1) To increase weather resistance through excluding actinic rays by use of opaque materials (graphite, lampblack, etc.), (2) to add color, and (3) to serve as an extender. For the inert filler, almost any finely ground, water-insoluble mineral matter is satisfactory. The more inert materials, such as mica, talc and slate dust are superior to lime, limestone or cement. Generally fillers with a rounded shape produce the best weathering characteristics when relatively fine. The best size grading must be determined for each particular mix. There appears to be one certain combination of kind, grading, and amount of filler, thickness of film and composition of asphalt that gives optimum resistance to weathering. The properties to be considered are: (1) chemical characteristics, particularly activity to moisture, asphaltic oils and dilute acids, (2) wettability, (3) particle shape, (4) particle size, (5) hardness and (6) porosity.

The National Bureau of Standards recently reported<sup>17</sup> results of a study of the effect of fillers on the weather resistance of asphalt roofing, using 57 different combinations of asphalts and fillers, including slate flour, dolomite, limestone, silica sand, silica dust, greenstone, hydrated lime, traprock, diatomite, mica and talc. The tests showed without doubt that fillers improved the weather resistance of the roofing. All fillers were not equally effective. Although not strictly a filler use, the same materials are used as dusting powders to prevent the roofing from sticking together after it is rolled for shipment. A small amount becomes embedded in the surface, but it is soon removed in handling or washed off shortly after laying. The use of coarse mineral granules in the surface layer is not within the field of this paper.

*Mixed Fertilizers.*—Fertilizer fillers differ from the others described here, in two respects: (1) They are sometimes added solely as diluents and (2) they are the only ones for which any statistics are available. Sand is the principal material used. Earth, cinders, coal ashes, sawdust, and almost any available granular inert can be and have been employed. Limestone, dolomite and phosphate rock are usually considered as fillers, although they may supply necessary minor plant-food elements such as lime and magnesia, or a major element in a relatively unavailable form (for example,  $P_2O_5$  from phosphate rock) or neutralize physiological acidity. Dolomite or finely ground phosphate rock certainly are worth more to the average consumer than sand. Cooper and Paden<sup>4</sup> have

shown that dolomite as fertilizer filler has been very beneficial to crops raised on acid soils in South Carolina. Its use is rapidly increasing and, if filler must be used, certainly it is the best material from the standpoint of the average consumer.

Mehring and Peterson<sup>12</sup> show the following amounts of filler (including all materials not supplying available N,  $P_2O_5$  and  $K_2O$ ) used in mixed fertilizers for the years mentioned:

Year	1900	1905	1909	1913	1917	1919	1925	1929	1931
Per cent . .	6 75	7.26	7 96	9 32	19.36	18.49	12.28	13 29	15.17

They estimate that over 1,000,000 tons of fillers was used in fertilizers in 1917, the maximum, and almost 700,000 tons in 1931.

In a survey of materials used in making mixed fertilizers in seven southern states (Alabama, Florida, Georgia, Mississippi, North Carolina, South Carolina and Virginia) in the year ending June 30, 1934, Mehring<sup>11</sup> found that 446,173 tons, or 17.96 per cent of the 2,484,237 tons of fertilizer sold, was filler. In North Carolina, the average filler content was 21.22 per cent. From the standpoint of the farmer this is an uneconomic practice and an educational campaign is being pursued to raise the grade of mixed fertilizer used. Mehring has figured that in the 1934 season, the southern farmer on the average paid \$2.21 for filler in each ton of fertilizer purchased, or \$12.47 per ton of filler, an expenditure of \$5,482,227, much of which was unnecessary. Fertilizers containing 20 per cent or more of total plant food (N,  $P_2O_5$  and  $K_2O$ ) contain little or no filler.

For many years, the use of filler in mixed fertilizers has been steadily increasing because the plant-food content of available materials has increased much more rapidly than that of the mixed goods demanded by the consumer. Many consumers still ask for grades containing only 14 or 16 per cent of total plant food, but the raw materials available at economic prices when combined in the usual proportions make grades containing 20 per cent or more. The grade of the raw materials is steadily being raised, and unless steps are taken to raise the average grade of mixed fertilizers sold even larger amounts of filler will be used in the future.

*Molded Products.*—Upon the proper selection and use of the filler, no less than of the binder, depend the qualities of finished molded products. Resins alone, when pressed or cast and cooled, develop internal strains that lead to cracking. Fillers minimize this shrinkage. Ellis<sup>6</sup> says:

While it is true that the cost of fillers on a volume basis is generally less than that of the resinous binder which holds them together, fillers should in no wise be considered adulterants. . . . Many valuable properties of molded articles, in addition to strength and shrinkage, depend on the selection of a filler and a binder adapted to each other and to the problem in hand.

The most universally used filler in molded compositions of the thermosetting type is wood flour. Asbestos is used extensively in compositions that are to withstand higher temperatures than can be resisted by wood flour or other organic fillers. Long-fiber grades give better strength than short-fiber. Mica alone, or with other inorganic fillers such as gypsum, may be used with shellac or phenol-sulphur chloride resins to make products containing 70 to 90 per cent by weight of filler. Fine inorganic materials such as china clay, gypsum, rottenstone, barite, slate dust, diatomite and quartz are used to some extent, but generally require the use of a reinforcing fibrous or lamellar filler if strength is not to be sacrificed. Phonograph records, for which hardness is more essential than strength, contain high proportions of these fillers. Whyte and Lord\* have incorporated up to 30 per cent of diatomite in synthetic resin or cellulose ester or ether films with a resulting increase of resistance to abrasion and cutting without serious loss of transparency.

*Paints.*—Fillers, known to the paint industry as extender pigments, are added to most ready mixed paints to prevent settling. In some they increase weather resistance and, for colored paints, tint retention. Also, the smaller cost than that of the prime pigment replaced cannot be overlooked. Ayers says<sup>2</sup> that diatomite can be used to flat a paint or lacquer without excess pigmentation. Often dead flats cannot be made except with diatomite unless an excessive amount of pigment is used. Calcium carbonate having a low oil-absorption property is added to white enamels to reduce the drag and improve the leveling of the enamel. Precipitated whiting, having high oil absorption, improves the dry hiding power of inside whites, and also aids in producing a dead flat effect. Ground limestone and precipitated whiting are both widely used in cold water, calcimine and casein-base paints.

Transparent, white, water-ground mica can be used in small amounts to overcome checking and cracking of exterior paints. Talc is used chiefly as a suspending agent; fibrous talc lengthens the life of extended paints. Barium sulphate is a widely used high-gravity extender. Although clays are not used extensively as extenders, they are employed where abnormally high consistency is desired with low pigmentation. They are used extensively in surfacer undercoats.

The paint industry does not use crystalline silica because it settles rapidly in low-viscosity suspending liquids and causes excessive wear on grinding equipment. The amorphous type is used extensively in paste fillers and paints, particularly where tooth and adhesion are desired, or in industrial outside paints. Because of its resistance to high temperatures, it is also used in heat-resistant paints. Bleached amorphous silica can be added to zinc, titanium and lead paints without discoloration.

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\* British Patent 384604 (1931).



Durability of outside paints formulated with white opaque pigments such as zinc, lead, titanium, antimony, etc., can be greatly improved if the extender pigments cover a wide range in particle size and are used only in such amounts that the hiding power of the paint will not be greatly affected. To relieve the points of stress and strain, and to fill the voids in the dried paint films, the shapes of the fillers used should range from fibrous and crystalline to flakelike.<sup>14</sup> As the film ages, the extender particles will take up the strain created by the shrinkage of the film and prevent cracking and deep checking of the paint.

The National Paint, Varnish and Lacquer Association has published a list of about 2400 pigments, including several hundred extenders, in the eighth edition of *Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors*, by H. A. Gardner.

*Paper.*—Sutermeister gives the following information regarding the advantages of fillers in paper (ref. 18, p. 352):

Nearly all classes of papers, except a few for special purposes, contain some mineral filling or loading material and unless it is used to an excessive extent it cannot be considered an adulterant. In fact, without some filler it is impossible to produce many of the grades which modern printing practices demand, since it fills up the interstices between the fibers and gives a better surface for process cuts and halftones, which are so largely used. It also makes the paper more opaque, improves the feel, enables it to take a better finish on calendering, and lessens friction in printing, all of which are of importance to the trade.

Much less filler is used in writing paper than in printing paper. When fillers

... are used in very large amounts, and the paper subjected to supercalendering, they are of great assistance in producing the effects desired in imitation coated papers. When a filler is used in large amount it quite seriously reduces the strength of the paper produced; hence when the strength of a paper is specified, and a filler is used, a better grade of fibrous stock must be employed and more care used in manufacturing than if no filler is used.\*

In selecting loading materials, several considerations must be kept in mind: (1) chemical composition, since any substance that is too water-soluble or that contains free acid or chlorine compounds, cannot be used; (2) necessity of very fine material of uniform consistency free from sand and grit; (3) color that will not interfere with the shade of the finished paper; (4) retention, (5) specific gravity, and (6) index of refraction, which influences the opacity of the paper. From 10 to 15 per cent of filler is a customary amount, although as much as 35 per cent is often used in so-called "Bible" papers.

Clay is the material most commonly used as a paper filler; others are talc, asbestine, calcium sulphate, barite, *blanc fixe*, calcium carbonate, titanium pigments and zinc pigments. When making colored papers in

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\* Ref. 18, p. 379.

which soluble dyes are used, a filler with the greatest absorptive capacity for the dye chosen must be selected. Sutermeister<sup>18</sup> has tabulated such data from a number of sources.

*Rubber.*—It is well known that the addition of certain finely divided powders to rubber improves the mechanical properties of the product, imparting increased toughness, tensile strength, hardness, compressive strength, resistance to steam and to abrasion, etc. The "active" or reinforcing powders increase resistance to abrasion, toughness, resistance to swelling, etc. Nonreinforcing or "inactive" fillers not only serve as diluents and cheapeners but increase the hardness of the compound, serve to facilitate tubing, calendering and other processing operations, et cetera.

Carbon black is the most important and zinc oxide the second most important of the active fillers. Certain clays also are widely used. While statistics are not available, one gathers the impression from the literature on rubber compounding that magnesium carbonate, although once an important filler for rubber, is now little used. Barite, chalk, diatomite and whiting are employed chiefly as nonreinforcers. Powdered glass and pumice, although inactive, are used in erasers for their abrasive action. Barite, diatomite and colloidal silica find application in acid-resistant mixes.

The important considerations, aside from cost, in selecting a filler for rubber are the fineness, ease of dispersion, color and freedom from constituents deleterious to rubber. The active or reinforcing fillers are very finely divided, usually all through 300 mesh.

Twiss<sup>19</sup> has shown that the strength and toughness of a rubber mix increase with increasing fineness of mineral filler. Weigand<sup>20</sup> found that there is an unquestionable relationship between the reinforcing action of a filler and its surface area.

Some fillers such as whiting are now available in a form in which the individual particles are coated with stearic acid to facilitate dispersion in rubber. Many rubber products are made to meet certain color requirements, hence fillers command a higher price when white than when off-color. The particle shape<sup>8,9,15</sup> and degree of wetting (adhesive force)<sup>10</sup> are likewise of importance.

Compounds of copper and manganese are very deleterious to rubber; rubber fillers must not contain more than 0.001 per cent of these elements. Water-soluble impurities, irrespective of composition, are objectionable in fillers for rubber goods that are subject to prolonged contact with water.

There has been more careful scientific study of the effect of fillers on rubber than in any other field. A review of this work and references to the literature are given by Memler.<sup>12</sup> A comprehensive list and description of rubber fillers is given in the annual encyclopedia on *The Rubber Industry*.<sup>15</sup> The interested person also should read the comprehensive

paper by Bachmann, Behre and Blankenstein,<sup>3</sup> and the book by Dawson and Parrett.<sup>5</sup>

*Cosmetics.*—A face powder must possess four properties: slip, covering power, absorbence and adhesiveness. Slip is imparted by talc or stearates; covering power by ZnO, MgO, TiO<sub>2</sub>, BaSO<sub>4</sub>, CaSO<sub>4</sub> or lithopone; absorbence by precipitated chalk, MgCO<sub>3</sub>, or kaolin; and adhesiveness by zinc, magnesium or organic stearates. Typical formulas are: (1) talc 62 per cent, ZnO 20 per cent, precipitated chalk 13 per cent and zinc stearate 5 per cent, or (2) talc 60 per cent, BaSO<sub>4</sub> 30 per cent, chalk 5 per cent and magnesium stearate 5 per cent.

For toothpastes or powders, materials must be neutral, nonpoisonous, noncorrosive, deodorant, refreshing, lastingly antiseptic and leave no unpleasant tastes. Of course, fillers have no part in the last four. Any white, chemically inert powder passing 200 mesh can be used. The most popular material is precipitated chalk. Other materials that are used include talc, tricalcium phosphate, diatomite, fuller's earth, kaolin, MgCO<sub>3</sub>, pumice, TiO<sub>2</sub>, SnO<sub>2</sub> and MgO.

Precipitated chalk and talc, with various chemicals, are widely used in foot powders. Fillers also are used in soaps. Certain colloidal clays serve both as detergents and mechanical aids in cleaning. The more abrasive materials are used in scouring soaps.

*Cement, Mortar and Concrete.*—Innumerable materials have been exploited for the integral waterproofing of Portland cement mortar and concrete. Among these are a number of the mineral fillers previously discussed—bentonite, clay, diatomite, fuller's earth, silica, talc, lime. Fillers also are added to bituminous cements for the same general purpose as in sheet asphalt and roofing. The materials used are the same.

*Scouring Powders and Polishes.*—A scouring powder consists of a uniform mixture of soap powder and an insoluble mild abrasive, such as pumice, volcanic ash, quartz, marble or feldspar. In polishes, the filler must be one that will not scratch or mar the finish on which it is used. For highly polished surfaces, soft materials such as chalk, rouge or talc are employed; for slightly rougher work, pumice, volcanic ash and diatomite.

*Insecticide Carriers.*—Pulverized materials, such as hydrated lime, powdered limestone, talc, clay, and many other materials, are used as carriers for insecticides. There are no recognized general specifications as to size, chemical properties or physical properties.

#### TREND IN FILLER RESEARCH

Some work is now being done on the alteration of the surface of finely pulverized materials with organic modifying agents. Clay fractions obtained by froth flotation have a markedly different behavior from the

original clay in spite of the fact that reagent consumption is low. A monomolecular film of a water-repelling chemical apparently can give a greasy texture to a clay. Wet-working and burning characteristics of clay can be controlled through modifying the physical properties of surfaces of clay particles. Pulverized material that tends to cake can be made free-flowing by treatment. Consider the possibilities from the use of antioxidants, water repellers, etc.! The field has hardly been touched. Here is where research can contribute the most to filler technology. The result should be better products that may entirely change the present picture.

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## CHAPTER XXVIII

### MINERAL PIGMENTS

BY HEWITT WILSON\*

**M**INERAL pigments give color, opacity or body to paint, stucco, plaster, mortar, cement, linoleum, rubber, or similar materials. They must be fine, substantially insoluble, and inert in the vehicle used. They are grouped as follows:

1. The natural mineral pigments, including ocher, umber, sienna, ground slate and shale, and other colored rocks that are merely taken from the ground and prepared by some simple purification or grinding treatment.

2. Pigments made directly from the ores, including burnt or calcined siennas, umbers, zinc oxide and sublimed white lead.

3. The chemically manufactured inorganic pigments, including white lead, lithopone, Venetian red, chrome yellow, Prussian blue, and the red and yellow iron oxide colors made from scrap iron or iron waste.

Combinations of these three classes are common, and other classifications based on color hue can be made, such as white, yellow, red, brown, green, blue, and black pigments. Natural mineral earth pigments contain, as their essential color constituents, one or more of the three oxides of iron—limonite ( $\text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}$ ), hematite ( $\text{Fe}_2\text{O}_3$ ), or, more rarely, magnetite ( $\text{Fe}_3\text{O}_4$ ).

#### NATURAL MINERAL PIGMENTS

*Yellow-brown.*—Yellow ocher is the most important natural mineral yellow and has been used since prehistoric times under various names, including Mineral Yellow, Roman Ocher or Earth, Chinese Yellow, etc. It consists of the yellow-brown form of limonite containing 17 per cent or more  $\text{Fe}_2\text{O}_3$ , with varying amounts of clay, silica and other impurities, which cause different chroma and brilliancy. Sienna, Italian earth, Terra di Siena, was found first near Siena, Italy. Ladoo classified it

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Based on an investigation made by the U. S. Bureau of Mines at its Northwest Experiment Station, Seattle, Wash., in cooperation with the College of Mines, University of Washington. Published by permission of the Director, U. S. Bureau of Mines.

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with the brown pigments as the iron oxide content (25 to 68 per cent) usually is greater than that of the average ocher, but recent studies indicate that no sharp division can be made between sienna and ocher in either hue or iron oxide content. The siennas grade into the average ocher with lower iron contents and into umbers with an increasing manganese dioxide content.

*Red and Red-brown.*—The hue of natural red pigments is derived from hematite. The ferric oxide content of such pigments may range from nearly 100 per cent in artificial reds down to about 5 per cent  $\text{Fe}_2\text{O}_3$  in calcined clays and shales. The most common natural red pigments on the market are Persian red and Spanish red. Persian red, the original Indian red, contains 65 to 72 per cent  $\text{Fe}_2\text{O}_3$  and is imported from Ormuz Island, in the Persian Gulf. The full-strength color is rich crimson and on dilution with white pigments develops a bluish tint. Spanish reds, containing 82 to 87 per cent  $\text{Fe}_2\text{O}_3$ , are nearly pure red hematites from Spain. Venetian reds were imported from Italy originally, but the term is applied now to the cheaper grades of diluted, artificial red pigments. Domestic reds, containing 10 to 60 per cent ferric oxide, are produced for service as base pigments for red paints, to which other red pigments are added to increase the chroma. Although numerous deposits of red clays, soils and rocks are found distributed over the country, their iron oxide content is insufficient to stand the dilution required for paint and pigment service. Hence, with few exceptions, the red iron oxide pigments are produced by roasting or calcining the more concentrated iron ores.

*Browns.*—The characteristic concentrated limonite color is a brown-black, which on dilution with white powder changes to a lighter brown or shows new hues, such as yellow-brown, greenish brown, or a neutral gray. The brown color is common when manganese dioxide is present with the iron oxide. The raw umbers, or terra ombra, are very dark brown-blacks, showing slight yellowish browns to rich greenish secondary hues when in the concentrated condition. On dilution they change to beaver, seal, or putty brown colors. Umbers contain 11 to 23 per cent  $\text{MnO}_2$ , with 25 to 47 per cent  $\text{Fe}_2\text{O}_3$  and often considerable carbonaceous material. The siennas, with their high iron oxide content, show stronger brown colors in the concentrated condition than do the yellower ochers, with the exception of the Italian sienna, which is quite yellow. On the other hand, the so-called "Georgia" ochers have more of the typical brown color of siennas. These are examples of the confusion in pigment nomenclature.

*Blacks.*—While the concentrated raw umbers approach black in hue, the true blacks, which on dilution reduce to a neutral gray, are made principally from some form of carbon with or without the black oxide of iron. The true carbon blacks are: (1) the soot blacks, including carbon

black, lampblack and gas black; (2) animal blacks, including ivory black, boneblack and dropblack; and (3) charcoal blacks. Mineral blacks and slate blacks are heavy black slates or shales that are used chiefly as a black filler for ironwork but often are toned with the carbon blacks because the original tinting strength is small. Powdered coal, manganese ores, graphite and asphaltum are used as black pigments, particularly the graphite.

*Green.*—Green earth, terre verte, green ocher, Celadon green and Verona green are the only important natural mineral greens. They are the ferromagnesium silicates, including soapstones, greenstones, and other rocks of dull greenish color, rich in chlorite, derived from pyroxene, amphibole, biotite and other minerals. They are used chiefly as a base for cheap lakes or bases for the artificial chrome and zinc greens, since their original tinting strength is low.

*Miscellaneous Colors.*—White clays, talc, whiting, dolomite, gypsum, barite and other natural white minerals can be used for white pigments in plasters, mortars, cold-water paints, calcimines, paper, etc., but cannot equal the opacity of white lead, zinc oxide and lithopone in the oil paints. They are used widely as fillers and extenders.

There are no natural mineral blues now in use, although lapis lazuli ( $3\text{Na}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 2\text{Na}_2\text{S}$ ) was highly valued as a deep blue pigment by the ancients. The mineral vivianite, a hydrous ferrous phosphate,  $\text{Fe}_3\text{P}_2\text{O}_8 \cdot 8\text{H}_2\text{O}$ , has a strong cobalt blue color. Small lumps of vivianite are found in clay deposits where a bone of some animal has come in contact with damp, ferruginous silicates.

#### PROPERTIES

*Color.*—Since color is the most important property of a pigment, the iron oxide of the natural mineral pigments must be in such condition that the color is pleasing and is not associated with the contaminating impurities that lower its esthetic value. The iron oxide yellows, reds and browns are the end-product colors derived by the weathering of all types of rock and therefore are very resistant to change during the lifetime of most structures. Those used by the early Egyptians have been discovered in their original bright, strong colors after centuries of burial or exposure to sunlight.

*Particle Size.*—The natural pigments consist of minute particles ranging from the colloidal clay and hydrated ferric oxide to those hard rocks which after grinding are just finer than 325 mesh, 200 mesh or even 100 mesh. Ayers<sup>3</sup> gives the average particle-size range of the natural earth colors from 0.85 micron for Persian Gulf red oxide to 2.20 for domestic red oxide, and the range for the synthetic colors as 0.35 micron for copperas red to 0.90 for Venetian red. The maximum percentage weight

<sup>3</sup> References are at the end of the chapter.



coarser than 325 mesh allowed by the American Society for Testing Materials for the red and brown iron oxide pigments is 3.0 per cent and only 1.0 per cent for ocher.

*Specific Gravity and Hardness.*—The true specific gravity of these powders ranges from that of the iron ores—magnetite (5.0), hematite (4.8 to 5.3) and limonite (3.6 to 4.0)—to that near clay (2.6). French ocher, with 20 per cent  $\text{Fe}_2\text{O}_3$ , is given a specific gravity of 2.88 by Ayers.<sup>3</sup> The bulk density of these powders ranges from 0.02331 to 0.04168 gal. per pound. The linseed-oil absorption ranges from 14.0 per cent for the coarsest particle size to 80 per cent for certain near-colloidal precipitated colors. The original hardness of the ores ranges from that of hard limonite or hematite to soft colored clays or gypsum. However, even the softest and purest china clays are composed of submicroscopic kaolinite particles plus quartz impurities that are harder than steel.

#### ORIGIN, MODE OF OCCURRENCE AND DISTRIBUTION OF DEPOSITS

Since the coloring power of the natural yellow, red, and brown mineral pigments is due principally to the content and condition of iron oxide, the occurrence of mineral pigments in many instances is closely allied to that of the iron ores. Pigment materials and iron ores often are mined in the same localities, or iron ores are used at times for mineral pigments of the red and brown varieties.

Replacement or precipitation deposits are the principal sources of limonite and ocherous minerals. They have been deposited in cavities by ground waters charged with iron salts removed from the weathering of impure limestone, sandstones and shales, especially when pyrite was an accessory mineral. The most important deposits are found usually in the fractured and faulted zones of rocks of all ages, including the Cambrian quartzites of Georgia, the Paleozoic limestones and quartzites of Pennsylvania, and the unconsolidated Tertiary clays, sands, manganese ores, and lignites of Vermont.

The chief production of ocher, sienna, umber, and similar iron oxide pigments in the United States comes from Pennsylvania and Georgia, with New York, California, Illinois, Tennessee, Virginia, New Jersey and Ohio producing smaller amounts.

From abroad, bright natural red hematite comes from Spain and the southern shores of the Caspian Sea. Deposits of the iron oxide, manganese ores having a brownish green hue, occur on the Island of Cyprus and are known in the United States as Turkey umbers. The Italian raw and burnt siennas are imported from Siena in northern Italy; the yellow ochers come from France, South Africa, and India. It is believed that all portions of the world containing ferruginous rocks that have undergone weathering have or will contribute to the mineral-pigment

industry, locally if not in the world markets. Iron oxides are universally distributed.

#### COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

The United States possesses all the varieties of iron oxide mineral pigments, but the natural ochers and red oxides mined at present do not have the brilliance of certain foreign pigments. (However, the development of the domestic synthetic pigments has successfully invaded the market for all products save possibly sienna and umber.) Many other natural pigments will be found in the near future, as indicated in a recent report.<sup>12</sup> The United States has wonderful resources for most of the raw materials used in the chemical pigments, including iron ore, lead, zinc, cadmium, selenium, china clay, whiting, talc, barite, some manganese, some titanium, a little uranium, only a small amount of the chromite used, but no cobalt or zirconium. Deposits of chromite are being developed in Montana and Washington.

The tariff on imports is 20 per cent for the iron oxide and hydroxide pigments, both natural and synthetic, with about 0.9 per cent added to the synthetic for antidumping. Crude, not ground, ochers, siennas and umbers take  $\frac{1}{8}$ ¢ per pound, and the ground or washed grades have a duty of \$7.50 per ton. The tariff on brown earths such as Vandyke Cassal earth is 25 per cent.

Since the United States Geological Survey discontinued the canvass of natural mineral pigment production in 1915 the records are incomplete. Data from the United States Bureau of Census and the United States Bureau of Foreign and Domestic Commerce are given in Figs. 1 and 2. While the data are incomplete, it is believed that the synthetic iron oxides gradually are assuming a position of greater importance in the American pigment industry.

#### PROSPECTING, EXPLORATION AND MINING

An unusual color is the principal indication of a mineral pigment in the field. The beautiful red, yellow, brown and green rocks and clays in canyons, hillsides and river valleys are, for the most part, already diluted with aluminum silicates by nature below the concentration required for commercial pigments. Very few deposits of colored rock, when powdered and mixed with 10 or 20 times their weight of zinc oxide or white lead, will produce more than a perceptible tint, because of the low concentration of iron oxide. Many such colored rocks, if used alone without dilution, could find a limited local service for mortars and some wall colors, but could not compete with the more concentrated commercial pigments already on the general market. Therefore, a prospector's equipment should include a small porcelain mortar and pestle for fine-grinding small samples of colored rocks mixed with at least 10 times their

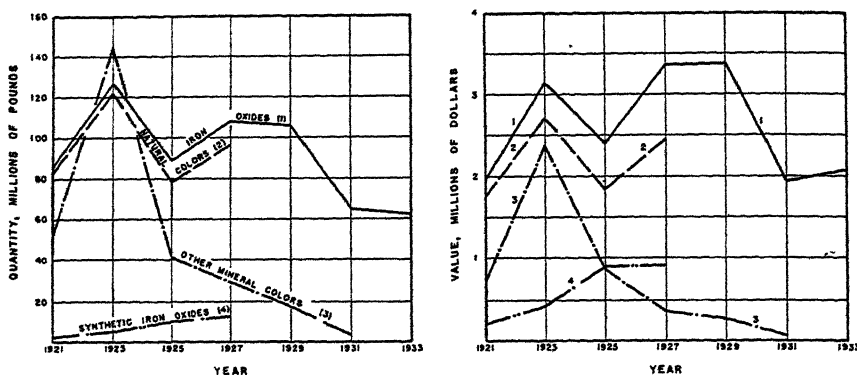


FIG. 1.—PRODUCTION AND VALUE OF IRON OXIDES AND OTHER MINERAL PIGMENTS IN THE UNITED STATES, 1921-1933.

Data compiled by the U. S. Bureau of Census.

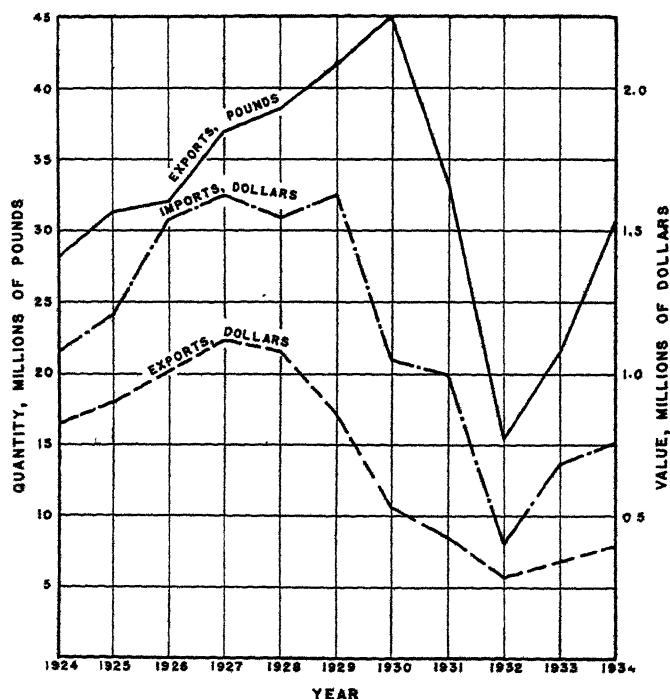


FIG. 2.—IMPORTS AND EXPORTS OF IRON OXIDE AND IRON HYDROXIDE PIGMENTS, 1924-1934, UNITED STATES.

Data compiled from Foreign Commerce and Navigation of the United States, U. S. Bureau of Foreign and Domestic Commerce.

weight of zinc oxide. These should be compared side by side with samples of various commercial pigments diluted in the same proportion in both the wet and dry condition. This direct comparison by persons with good color sense is necessary, as very few people can memorize accurately the different color hues, values, and chroma.

In Pennsylvania and Georgia the ocher is mined both by open-cut methods and by shafts. The former method is employed whenever ocher lies within 10 to 15 ft. of the surface, and shafts are sunk several hundred feet where it lies deeper. In most deposits the pockety character of the ore and the uncertain market for the product do not justify elaborate equipment. Typical mining operations are described in references 30 and 32.

The soft, claylike pigments are treated by comparatively simple washing processes, followed by dehydration and pulverization. Log washers and blungers are used for dispersion; trough, cone and bowl classifiers separate the sand from the fine suspension; and a portion of the water is removed in settling tanks while the remainder is extracted by filter presses and rotary driers. Pulverizers of the hammer type reduce the pigment to powder for packing and shipment.

#### ARTIFICIAL MINERAL PIGMENTS

*Artificial Red Iron Oxide Pigments.*—The first artificial pigments undoubtedly were the reds produced by calcining or roasting ochers, siennas or iron ores to red heat. Burnt ochers, burnt siennas, burnt umbers, red metallic paints, Canadian red, and others are examples of such calcination, which not only changes the darker brown forms of iron oxide to the bright red hematite but in lightly calcined umbers will cause any gray, carbonaceous matter to darken toward black unless overfired. Vertical shaft kilns, rotary furnaces, platform calciners or floor kilns are used. The next step duplicated the deeper crimson Persian or original Indian red by roasting purer forms of iron, such as byproduct copperas (iron sulphate), to produce Indian red, colcothar, rouge and crocus. The red ferric oxide produced by calcination may be diluted with white clay, powdered silica, and other cheap white diluents for the cheaper grades of Indian red and red oxide, with aniline colors for Tuscan red and with calcium sulphate for Venetian red. The latter also is made by neutralizing ferrous sulphate with lime. Dissolving scrap iron in sulphuric acid is another method of producing copperas for roasted or the chemically precipitated colors.

*Chemical Inorganic Pigments.*—Red, yellow, brown, black and their intermediate hues can be produced from iron salts, such as ferrous sulphate or ferric chloride, by varying the precipitation agent, the temperature and concentration. Artificial brown pigments are made by calcining certain siennas, umbers and limonite iron ores, especially those that

contain manganese dioxide, such as the Cappagh brown from Ireland. The lead compounds used as pigments are the red minium oxide, the red, orange and yellow lead chromates, the yellow massicot, the orange litharge and the white basic carbonate. Chromium oxide produces the most stable green pigment while the hydrated chromium oxide, Guignet green, is more brilliant and transparent. Cadmium sulphide is used for red, yellow and orange and zinc yellow is a complex salt of zinc and potassium chromates. Blue pigments are made from melted cobalt silicate and almost any other color can be produced from silicate glasses containing the proper metallic oxides. Other blues are made from iron ferrocyanide and iron and cyanogen compounds. Artificial graphite, carbon black, bone black, together with the ferroferric oxide, are used for black pigments. Most white pigments contain white lead, the basic lead sulphate, zinc oxide, lithopone or titanium oxide. More detail of the chemical pigments can be secured from Ayers.<sup>3</sup>

#### TESTS AND SPECIFICATIONS

*Mass Color.*—Accurate color analyses that indicate the percentage reflection or luminosity for each wave length can be made by spectrophotometers, colorimeters, photometers, and other color analyzers, and the curves can be plotted.<sup>12</sup> The mass color is determined roughly by making a comparison of the unknown pigment with some approved color chart or system.<sup>17</sup> It is common practice to use pastes of the pigment in clear, refined linseed oil for paints, but the medium that is used in commercial practice should be used for testing. A.S.T.M. specification D 387-34 T requires a specified number of “rubs” and “pickups” of measured quantities of pigment and liquid medium before the unknown paste is compared with some approved standard.

*Tinting Strength.*—Tinting strength or the power of coloring likewise is commonly determined by comparison with a standard pigment. The two pastes are compared side by side after a thorough mixing of each with the same amount of standard white, zinc oxide, oil paste.<sup>2</sup> Additional refinement brings both pastes to the same consistency. Quantitative results can be obtained by varying the amount of pigment to zinc oxide until the color strength of the standard is matched, or vice versa. The ratio of the amount of the unknown pigment to that of the standard is then taken as the numerical value for the tinting strength of the unknown. The percentage gain in luminosity, measured by a tintometer or reflectometer, when a definite weight of zinc oxide is added, also can be used for numerical values of tinting strength.

*Particle Size.*—The maximum allowable percentage of grit determined by washing with water or kerosene<sup>19,20</sup> ranges in various specifications from 0.0 per cent coarser than a 200-mesh sieve to 3.0 per cent coarser than 325-mesh. Grit can be detected by the teeth, which are very sensi-

tive to hard, sandy particles. The particle sizes that are important for tinting strength are finer than the finest sieve openings, and special tests that have not been accepted as standards are necessary for this determination. Such methods include water elutriation,<sup>31</sup> sedimentation, air flotation,<sup>28</sup> and microscopic<sup>13,21</sup> and opacity determinations. The tinting-strength test is an indirect method for testing fineness.

*Oil Absorption.*—The particle size, together with the character of the surface and the possible porosity of the particle itself, affects not only the color and tinting strength but also the amount of linseed oil or other expensive liquid medium required to produce a given degree of fluidity or viscosity. Several hand-mixing methods have been devised to determine the amount of oil required under controlled conditions. The linseed-oil requirements to produce pastes of equal spreading consistency range as follows for a number of commercial pigments: French ochers, 30 to 37 per cent; American ochers, 35 to 53 per cent; raw sienna, 25 to 70 per cent; raw umbers, 44 to 53 per cent; yellow oxides, 29 to 49 per cent; red oxides, 14 to 28 per cent.

*Opacity and Hiding Power.*—The property that enables a paint or pigment to obliterate the background or to reflect the incident light before it reaches the background varies with the total pigment surface in a given area of paint and the difference in refractive indices of the oil and pigment. As a rule the hiding power varies inversely with the particle size to a certain fineness beyond which a loss of opacity is shown by certain pigments. Some of the refractive indices of the common white extenders and opacifiers are as follows: whiting, 1.49; quartz, 1.55; gypsum, 1.59; barium sulphate, 1.64; lithopone, 1.84; white lead, 2.02; antimony oxide, 2.09 to 2.29; zinc sulphide, 2.37; titanium oxide, 2.76. Their relative hiding powers have been determined as follows: white lead ground in linseed oil, 100; titanox B XX, 167; zinc oxide, 103; lithopone, 85; basic lead sulphate, 82; whiting, 5; china clay, 4; asbestine (talc), 2; barite, 1; and gypsum, 1. Hiding-power tests described by Gardner<sup>12</sup> include the use of cryptometers, hidimeters, photometers, colorimeters, and simulative service brushing tests over colored backgrounds.

Miscellaneous physical tests include: (1) specific gravity, determined by the usual pycnometer methods for powders but which is of little value except for general comparisons; (2) settling characteristics of the pigments with water or oils as fluids or pastes; (3) light and ultraviolet resistance; (4) weather-exposure tests; (5) insulation values; (6) the wetting properties of pigments in various liquids; and (7) high-temperature tests.

*Chemical Composition.*—The usual chemical analysis of mineral pigments, giving the percentage weights of silica, alumina, iron oxide, lime, magnesia, alkalis and ignition loss, indicates the total quantity of iron oxide, the accompanying coloring ingredients, and the type of base,

such as clay or gypsum, but does not give definite information about the original color, the mineral constituents of the diluting base material, or the important physical characteristics, such as color, fineness of grain, workability and degree of iron oxide dispersion. The chemical analysis can be used in standardization tests for specific pigments to maintain certain minimum requirements for the chief coloring ingredients and it will also reveal objectionable materials. An accurate and complete analysis, however, is expensive and difficult, whereas practical physical tests are easy and require little time.

The very general nature of the requirements found in the Government and private specifications indicates the minor importance of chemical analyses in comparison with physical tests. The United States Government and the American Society for Testing Materials require a minimum  $\text{Fe}_2\text{O}_3$  content of 17.0 per cent for ocher, a maximum of 5.0 per cent  $\text{CaO}$ , and no organic coloring matter or lead chromate. For red and brown oxides and iron hydroxides the same agencies require a minimum of 30 per cent  $\text{Fe}_2\text{O}_3$ .

The average ferrous oxide content for 49 miscellaneous ochers and earthy pigments in a recent study<sup>32</sup> was 0.04 per cent and no correlation could be made between the ferrous iron oxide content and the color. With pigments of the same hue and similar origin and geologic history there may be a relationship between tinting strength and content of ferric oxide. When natural pigments of different geological history are compared, however, wide discrepancies are found.\*

#### MARKETING, USES AND PRICE

The lack of accurate color and hue standards for mineral pigments has resulted in considerable confusion in nomenclature. This is noted especially among the large variety of colors introduced in the market from American deposits to duplicate some well-known European pigment but differing widely from that for which it was named. This practice not only discredits the American pigments but shows a lack of originality in devising new names and in marketing a useful material. Ladoo<sup>16</sup> states:

Domestic mineral pigments usually are either mined by some of the larger consumers, or marketed by the producers directly to the consumers. There are only a few large producers in this country, and they in a certain degree control the market for domestic mineral pigments. These producers usually handle a wide variety of natural colors and have built up reputations for uniformity and high grade of products. Many large customers prefer to deal only with such companies rather than to buy each color from a different small producer. Some of the smaller producers market their product through brokers or

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\* The American Society for Testing Materials gives standard methods such as the following: For red lead, D 49-33; chromium compounds, D 126-27; titanium-barium, D 382-34 T; titanium-calcium, D 383-34 T; titanium dioxide D 384-34 T; high zinc sulphide lithopone, D 385-34 T; and zinc sulphide, D 386-34 T, etc.

jobbers. Imported pigments are more generally handled through large importers, chiefly in New York, but some consumers import part of their pigments directly.

The prices of most natural mineral pigments have decreased within the last 10 years. The following are typical illustrations taken from the *Oil, Paint and Drug Reporter*, the last quotations being from the issue of May 11, 1936: American golden ocher, quality A, has dropped from 8¢ per pound in 1926 to 6¼¢ in 1936; French light ocher, from 4⅝¢ in 1926 to 3.0¢; natural yellow oxide, from 6¼¢ in 1926 to 4.5¢; synthetic yellow oxide, from 12¢ in 1929 to 6¾¢ now; American Indian red, 80 to 85 per cent, from 9¢ in 1926 to 7¢; pure American Indian red, from 15¢ in 1926 to 8¾¢; metallic red from 2¼¢ in 1933 to 2.0¢; pure red oxide from 14¢ in 1932 to 8.5¢; Persian Gulf red from 5¢ in 1931 to 4⅝¢; Spanish red oxide from 4.5¢ in 1929 to 3¼¢; Venetian red, from 5¢ in 1932 to 3¾¢; American raw sienna from 4¢ in 1925 to 2⅝¢; Italian raw sienna, grade 1, is now at the maximum of 12.5¢; American burnt sienna from 4¢ in 1932 to 3¢; Italian burnt sienna from 12.5¢ in 1930 to 12¢ for grade 1; American umber from 4¢ in 1927 to 2⅞¢ now; Turkish umber from 6¢ in 1929 to 4¢; and metallic browns from 2.5¢ in 1925 to 2.0¢. These are New York prices in large lots. The highest prices are the maxima since 1925. The best American yellow ocher is an exception, since it shows a rise from 2.0¢ in 1925 to 2¾¢ at present. French dark ocher has maintained a fairly even price near 3.0¢ since 1925, although it was as low as 2.1¢ in 1932.

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## CHAPTER XXIX

### MINOR INDUSTRIAL MINERALS

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#### ALUM MINERALS

THE alums comprise a series of double sulphates (or selenates) isomorphous with potash alum which, according to a ruling of the Pennsylvania courts, is the only product commercially described simply as "alum." Common alum occurs in nature as kalinite  $[K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O]$ ; the mineral mendozite  $[Na_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O]$  is soda alum; tschermigite  $[(NH_4)_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O]$  is ammonium alum; and there are about a dozen other native alums and related double sulphates. Alunite (alumstone or alum rock) has the formula  $Al_2(SO_4)_3 \cdot K_2SO_4 \cdot 4Al(OH)_3$  and alunogen,  $Al_2(SO_4)_3 \cdot 18H_2O$ . Saline residues found wherever natural water is evaporated may contain sulphate of alumina, which for most uses is the essential ingredient of alum. Mineral wells and springs that emerge from beds of pyritiferous shale are typically astringent and at times form local deposits of alum, generally as incrustations. Acid waters acting upon aluminous rocks likewise may form sulphates of alumina.

The alums of commerce, as well as aluminum sulphate (concentrated alum) are produced principally by treating bauxite with acid, but they have also been made on a substantial scale from clay, cryolite, pyritic slates, schists, lignites, alunite and leucite. Alum shale—chiefly clay, pyrite and (usually) carbonaceous matter—was extensively used in England as a source of alum for over two centuries; and in France, pyritic lignites mixed with sand and clay were burned in heaps to red ash, the leach liquors from which, after treatment with ammonia, yielded alum and copperas. Processes have been developed for making alum or aluminum sulphate from such diverse materials as feldspar, aluminous phosphate minerals, greensand, and mixtures of mica and potash salts or of clay and alkali sulphates—most commonly by baking the material with sulphuric acid at low red heat.

*Kalinite* occurs in an irregular network of veins associated with sulphur in rhyolite in Esmeralda County, Nevada. At a recovery plant operated there between 1920 and 1923, the ore was crushed to  $\frac{3}{4}$  in., ground to

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pass about 80 mesh, and leached (countercurrent); the resulting alum solution was then filtered and crystallized. Kalinite-bearing rocks are occasionally baked with acid to increase the yield, and if the natural liquor does not contain enough potash to form potassium alum, potassium sulphate may be added.

*Alunite* has received a great deal of attention as a possible source of potash. Deposits are found in several western states—Utah, Colorado, Nevada, Arizona and California—as well as abroad. In recent years small quantities of alunite have been mined at Marysvale, Utah, and at Sulphur, Nev. Some of it has been used finely ground for direct application to the soil as fertilizer. During the World War, it was used as raw material for potassium sulphate, and in Europe alunite has been used more or less extensively for making potash alum. Although insoluble in its natural state, the mineral is readily decomposed into  $K_2SO_4$  and  $Al_2O_3$  by heating to above  $750^\circ C$ . At present, alunite deposits are receiving renewed consideration as a possible source of alumina for the manufacture of aluminum.

*Production and Consumption.*—Alum was being produced commercially in the United States as early as 1815, the domestic industry receiving its first impetus from the cutting off of imports during the War of 1812. Alum-making from bauxite was begun in 1877 and for 1880 the Census reported that six establishments produced 19,600 tons of alums valued at \$808,165. In recent years the domestic output of alums, chiefly soda alum, has averaged a little less than 25,000 tons a year, a relatively small increase during the last half century. The production of aluminum sulphate, however, advanced to 364,000 tons in 1935. Imports are small, averaging scarcely more than 1000 tons annually, whereas exports (almost wholly aluminum sulphate to Canada) exceeded 33,000 tons in 1935. Over 150,000 tons of bauxite, much of it imported, is used annually in the United States for the manufacture of alums and aluminum salts.

*Markets and Prices.*—Aluminum sulphate generally is marketed in two grades, both containing about 45 per cent combined water and approximately 37 per cent  $SO_3$ : (1) iron-free (under 0.1 per cent  $Fe_2O_3$ ); and (2) commercial. The theoretical composition calls for 18 molecules of water, but this product tends to be sticky, so the commercial product may be evaporated to a content of about  $14H_2O$  and, for at least one important use, down to  $7H_2O$ . A very small quantity of anhydrous sulphate is sold. Aluminum sulphate is mostly crushed and bagged or barreled. The usual container is a 200-lb. jute bag, though paper-lined bags have been used to some extent and experiments are being made with 100-lb. paper bags. The alums are available in various technical grades (lump or powdered) and also as C.P. crystals and as U.S.P. crystals, granules and powder. Sodium-aluminum sulphate, however, is marketed commercially only in anhydrous condition, generally ground. So-called

"porous alum" is ordinary aluminum sulphate to which a very small amount of soda ash was added just before solidification, thereby liberating enough carbon dioxide to fluff it up and thus make it more quickly soluble; as the ground product is being more generally used than lump, porous alum is of less importance than formerly.

A leading use of potash alum is for treating furs, though it has some medicinal and special uses. Ammonium alum is used, largely in lump form, for making statuettes and other cast-stone or stucco products, and it may be employed instead of potash alum for sundry purposes. Sodium-aluminum sulphate (not soda alum) is almost exclusively sold to baking-powder manufacturers, who buy it ground.

As mordants in dyeing, for making lake pigments, for the dressing of skins ("tawing") to produce white leather, and in the clarification of water, sewage, and turbid liquids generally, aluminum sulphate has displaced the true alums almost completely. Water purification and paper account for much of the consumption of this material, and surprisingly large quantities are used in fire-fighting compounds. There are many miscellaneous uses—such as dyeing textiles, tanning leather, and decolorizing or deodorizing mineral oils—but these account for scarcely 5 per cent of the total consumption.

Prices on the alums and aluminum sulphate do not change frequently. *Oil, Paint and Drug Reporter* quotations have remained about the same week after week for years. In 1936 the New York price of iron-free aluminum sulphate was quoted at \$2 per 100 lb., and for the commercial grade the price of \$1.35 had remained unchanged since 1933. For ammonia and potash alums, prices start at around \$3, the latter generally (though by no means always) being slightly the higher. Sodium-aluminum sulphate or so-called "soda alum" is in about this same price range or only a trifle higher; although it contains much more sulphur trioxide, its other ingredients are cheaper.

### BROMINE

Bromine ( $\text{Br}_2$ ), a dark, reddish brown, highly corrosive liquid, does not occur uncombined in nature but is widely distributed in the form of bromides, usually accompanying common salt and magnesium chlorides. Commercial sources in the United States include brine wells in California, Michigan, North Carolina, Ohio, Pennsylvania and West Virginia and also sea water. For many years, the world's bromine came principally from the potash deposits in the Stassfurt region, Germany. France began to produce bromine following return to their country of the Alsatian potash fields, but French producers have an agreement with the German cartel similar to that covering potash. Palestine is an important factor in the European bromine situation, the Dead Sea product being used extensively in England, which formerly imported bromine from the

Continent. Italy, Japan and Russia are in production and all salt-producing countries are potential suppliers. During the World War bromine was taken from brine wells in Tunis.

*Production.*—Although bromine was produced in the United States as early as 1846, there was no real demand for it until 1860, and the industry virtually began in 1865 with the recovery of less than 2000 lb. from the Stassfurt deposits. A year or two later, saltmakers in the Pomeroy district, Ohio, began to recover bromine, and domestic production grew to over a million pounds at the beginning of the twentieth century, only to drop to a quarter of that figure in 1910 as a result of competition from Germany, which had already produced more than two million pounds in a single year. Wartime needs of the United States and its allies boosted domestic output to 1,727,156 lb. in 1918, chiefly as sodium bromide. After 1919 there was some recession but since 1923, owing to increasing use of bromine in connection with the manufacture of antiknock gasoline, production in the United States advanced to 6,414,620 lb. in 1929 and jumped to 16,428,535 lb. in 1935. The larger part of the output is not sold as bromine but as ethylene dibromide, potassium and sodium bromide, and other compounds. Imports of ethylene dibromide exceeded 3,000,000 lb. in 1930 but amounted to only 477,005 lb. in 1935, and imports of other bromine compounds were negligible in that year.

*Technology.*—At Kure Beach, near Wilmington, N. C., is the only plant at which bromine is produced as a main product. Sea water contains only about one pound of bromine in 2000 gal., but huge quantities are pumped through a series of operations that includes a blowing-out tower wherein the bromine, previously liberated by acidification of the water and oxidation with chlorine, is blown out by compressed air. The sea water remains in the plant undergoing chemical treatment for scarcely a minute. The bromine is recovered in large absorption towers using soda-ash solution, and the resulting mixture of sodium bromide-bromate is treated with acid to liberate bromine again for use in making ethylene dibromide.

Elsewhere, bromine is produced from natural or artificial brines, usually from the concentrated bitterns remaining after the sodium chloride has been recovered. Recovery is commercially accomplished by one of three distinct methods: (1) a batch process using sulphuric acid and an oxidizing agent (such as sodium chlorate or manganese dioxide); (2) a continuous process involving treatment with chlorine gas; and (3) another continuous process in which an electric current instead of chlorine is employed to liberate the bromine.

*Markets and Prices.*—The recovery of bromine from the Stassfurt deposits, begun in 1865, soon brought the price down from \$10 to \$4.50 a pound and within the next few years to \$1.30. By 1885, it was 7¢ a

pound (0.70 mark per kilogram) in Germany and 25¢ a pound in the United States. In 1913 the wholesale price of bulk bromine in New York ranged from 25¢ to 35¢ a pound. After reaching the peak of \$6.50 a pound in 1916, the wartime price dropped to around 55¢. From 1926 until March 1931 quotations remained unchanged at 45¢ to 47¢, and from March 1931 through October 1936 it remained at 36¢ to 38¢. The average unit value of domestic output, f.o.b. plant or shipping point, in 1935 was 21¢ a pound, substantially less than the quoted price. This average rose from 13¢ in 1910 to a maximum of \$1.31 in 1916 and dropped to 15¢ in 1922, after which it rose to 34¢ in 1926 and declined to 21¢ in 1931, continuing at approximately that figure through 1935.

The principal use of bromine in the United States at present is as ethylene dibromide for making tetraethyl lead antiknock compounds, largely an integrated operation conducted by the Ethyl-Dow Corporation, in conjunction with the Ethyl Gasoline Corporation and the Dow Chemical Co. Outside of this enterprise liquid bromine is consumed largely for dye, photographic and pharmaceutical manufacture by the bromine producers and other companies, and is packed for shipment in cases containing nine 6½-lb. bottles. For use as analytical laboratory reagent, a small though important use, it is available in 1-lb. bottles. Bromides, other than ethylene dibromide used for making ethyl fluid, are sold to large chemical manufacturing organizations in bulk containers weighing 100 to 500 lb. net. These chemical manufacturing organizations either consume these bromides by compounding with other products to make pharmaceuticals or repackage in small containers for marketing through retail drug channels.

Previous to about 1923, bromine was used principally in the form of sodium, potassium or ammonium bromide in photography and for medicinal purposes. The substantial revival in demand after the Armistice came from the motion-picture industry, and there is a steady consumption for medicinal purposes, notably the treatment of nervous diseases. It is used for making fluorescein, eosin, Hoffman's blue, brom indigos, and other organic coloring matters for use in dyeing. During the World War some hand grenades and gas bombs were loaded with bromine or with organic bromine compounds. Brombenzyl cyanide, a tear gas for use in the trenches, was invented in 1917. "Mining salt," a sodium bromate-bromide mixture, was formerly used in some quantity for the extraction of gold from its ores, but this use seems to have virtually disappeared. However, acetylene tetrabromide has possibilities in the mining field in heavy solutions for separating ores; its use has been reported in South Africa for recovering diamonds and experimental plants have been operated in this country on various ores (Nagelvoort process). It is used in the automobile industry for oil and gasoline gauges.

Lower prices from bromine and the knowledge that inexhaustible supplies are obtainable from the ocean have stimulated research looking toward a larger use of bromine for organic syntheses and in metallurgy and some progress has already been made towards its use for water sterilizing.

#### CALCIUM CHLORIDE

Calcium chloride ( $\text{CaCl}_2$ ) occurs as an original constituent of natural brine and is produced commercially in connection with the extraction of salt or of salt and bromine. The calcium chloride thus recovered ordinarily contains a substantial admixture of magnesium chloride and small amounts of other salts, and for certain uses this naturally mixed calcium-magnesium chloride need not be further purified or separated. The principal commercial source of calcium chloride is as a byproduct of the Solvay process for the manufacture of sodium carbonate, the chlorine content in such case being derived from brine or rock salt but the calcium coming from limestone. In Germany, in addition to being a by-product of the Solvay process, it is recovered by the potash companies from carnallite ( $\text{KCl} \cdot \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ), which often carries as much as 3 per cent  $\text{CaCl}_2$ . Calcium chloride is actually listed as a distinct mineral, hydrophilite or chlorocalcite, occurring as a crystalline or mealy incrustation in the neighborhood of active volcanoes and, rarely, elsewhere, as an impure slimy exudation from sandstone, or mixed with clay. The hydrous calcium chloride ( $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ ) is known only in artificial crystals (hexagonal). More common are mixtures with magnesium chloride and other alkaline chlorides, notably tachydrate ( $\text{CaCl}_2 \cdot 2\text{MgCl}_2 \cdot 12\text{H}_2\text{O}$ ). Fluorspar ( $\text{CaF}_2$ ) contains chlorine in only minute quantities.

Anhydrous calcium chloride is a white, deliquescent substance that fuses at a red heat and, on cooling, solidifies to a translucent mass of cubic crystals of specific gravity 2.2. Its characteristic property is its avidity for water. Even crystals of the hydrated chloride ( $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ ) deliquesce rapidly, dissolving in only one-fourth their weight of water at room temperatures. A fused product may be purchased in C.P., U.S.P., and technical grades, but the principal commercial grades are: (1) flake (77 to 80 per cent); solid (73 to 75 per cent); and liquid (40 to 45 per cent).

The domestic production of calcium chloride almost trebled in value and more than trebled in quantity between 1923 and 1929, reaching a peak in the latter year of 277,010 short tons having an average value of \$21.47 a ton (converted to 73 to 79 per cent basis). In 1929 the output from natural brines as reported by the U. S. Bureau of Mines was 114,240 tons valued at \$18.36 a ton. In 1936, flake chloride cost in carload quantities (minimum,  $22\frac{1}{2}$  tons) from \$19 to \$35 a ton, delivered, the average for leading markets being probably around \$23. Solid calcium chloride is quoted at \$2 a ton less than flake. Liquid is sold f.o.b. works,

but freight may be equalized in competitive market territory; in 1936 the quotation of \$7.50 represented a saving of over \$2 a ton as compared with an equivalent amount of flake chloride.

The principal use of calcium chloride is for laying dust on highways and in making stabilized roads, leading buyers being the State and local Government highway boards. Ice control on highways and sidewalks and also for use by railroad and bus companies ranks second in importance, circulating brines at refrigerating plants (formerly the only large commercial outlet) now coming third. Next in order comes the treatment of coal and coke and sundry other dustproofing uses. Among miscellaneous uses may be mentioned curing concrete, fire buckets, and preserving the freshness of oak leaves and natural foliage. Calcium chloride is used as antifreeze in cooling condensers in the oil and gas fields but is not recommended for automobile radiators; although not corrosive of itself, it may set up electrolytic attack where two or more metals are in contact. As a dehydrant it finds a variety of minor applications; in addition to being the standard laboratory desiccant, it is used by oil companies to prevent internal corrosion of pipe lines, by gas works for drying gas, and in several industries for air conditioning. Ultimately a substantial quantity may be distributed by janitor supply houses for sale in small packages for removing or "skidproofing" ice on steps and walks. About 10 tons of calcium chloride may be used per mile of concrete roadway, but this use, especially on Federal-aid projects, has suffered from the introduction of cotton mats.

Inasmuch as highway construction and upkeep account for so large a proportion of the consumption of calcium chloride, it would be natural to conclude that sales should be distributed geographically more or less in proportion to population and road mileage, but this is not true at present. Michigan uses much larger quantities than one might expect, and there is an abnormally high consumption also in parts of Ohio and Indiana and in the vicinity of Chicago, although the use of calcium chloride (as well as of its competitor, sodium chloride) is spreading to all states, even in localities remote from producing centers where freight rates add greatly to the cost.

#### EPSOMITE AND OTHER NATURAL MAGNESIUM SALTS

Epsomite ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) is deposited from spring waters as at Epsom in Surrey, England, and has been mined near Oroville, Okanogan County, Wash., and elsewhere, although never to any large extent. Kieserite ( $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ ), which is found chiefly at Stassfurt, Germany, and Hallstatt and Kalusz, Galicia and which can be readily separated from associated saline minerals because of its relatively slow solubility, is used as raw material for epsom salts, which are also made from magnesite and dolomite.



According to Census reports, domestic production of epsom salts in 1935 was 38,489 tons valued at \$1,116,533, and imports were 1,530 tons. Kieserite was formerly imported in substantial quantities from Germany, but, notwithstanding the fact that it is specifically free of duty under the Tariff Act of 1930, few shipments have been reported in recent years. Imports of epsom salts, likewise, have diminished greatly and now are confined largely to Norfolk, Va., where they are distributed to tobacco growers as fertilizer. In 1935, domestic production of epsom salt from natural sources alone rose to 30,246 tons, and the total production of magnesium salts (sulphate, chloride, and carbonate) from sea water and natural deposits, brines, and bitters was 53,813 tons.

### IODINE

Iodine ( $I_2$ ) is a gray or purple-black, crystalline solid, semimetallic in appearance and diffusing a chlorinelike odor. The vapor is one of the heaviest known gases, 8.8 times as heavy as air, and its characteristic color gave the element its name, which is derived from the Greek word for "violet." Solid iodine (sp. gr. 4.7) is the heaviest nonmetallic element. In chemical affinity it resembles other members of the halogen group although it is markedly less energetic, and because of its high atomic weight (126.9) much larger quantities are required for a given reaction. Though almost insoluble in pure water, iodine is very soluble in solutions of potassium iodide, owing to formation of a compound,  $KI_3$ .

In minute quantities, iodine is widely distributed throughout the animal, vegetable and mineral kingdoms. It is always present in sea water but not in sufficiently large amounts to offer promise for its direct commercial recovery. A few varieties of seaweeds, however, are able selectively to absorb the element within their cell structure, especially at certain seasons of the year, and a good deal of iodine has been recovered from the ash or "kelp" obtained by carefully burning such weeds as were harvested along the stormswept coasts of Ireland, Scotland, France, Norway, Russia and Japan. The bulk of the world's iodine, however, has come as a by-product from the working of the natural nitrate deposits in Chile, where the iodate minerals, dietzeite and lautarite, have been identified in the *caliche*. In Java salt wells yielding 0.1 gram or more of iodine per liter have been worked for iodine since before 1900, and beginning about 1925 Italy has developed a small industry based upon brine wells at Salsomaggiore. Production from oil-well brines began in California and Louisiana in 1928 and by 1932 reached commercial proportions, and similar sources have been developed in Russia, Poland and Mexico. Iodine in small quantities has been found in various sedimentary deposits, especially coal, and a potentially commercial concentration of iodine (0.1 to 0.3 per cent) in Cottrell precipitator flue dust

at an iron blast furnace in Czechoslovakia was traced back in large part to the coke.

*Production.*—The output of iodine is determined by world demand and has increased rather slowly. Present-day requirements of around 1000 tons annually are only about three times the quantity sold in 1887. Immediately before the outbreak of the World War production averaged about 500 tons, and a maximum output of 1500 tons was reached in 1915. Chile normally furnishes 70 per cent or more of the total supply and could easily increase its production several fold.

Except for an insignificant quantity recovered in California as a result of wartime experiments with seaweed, the United States, although consuming about 30 per cent of world consumption, produced no iodine until 1928. By 1933, three plants were extracting iodine from waste waters of certain deep oil wells in the Long Beach field in California, and their sales were 401,525 lb. Imports, which amounted to only 278,713 lb. in 1931 and 631,669 lb. in 1932, jumped to 1,411,687 lb. in 1933 and 1,481,123 lb. in 1934, while domestic sales dropped. In 1935, imports were 375,819 lb. and domestic sales 245,696 lb. These figures indicate a consumption approximating the average imports of 646,437 lb. a year during the five-year period, 1925–1929, but any calculations based upon international trade figures are likely to be in error on account of the huge stocks of Chilean iodine that have been accumulated in several consuming countries, including the United States.

*Technology.*—The old method for recovering iodine in Chile was by precipitation with  $\text{NaHSO}_3$  but various other precipitants may be employed, including direct gassing with  $\text{SO}_2$ . In Java, the iodine is recovered in the form of insoluble cuprous iodide. In the French seaweed industry, 25 tons of weed shrink to 5 tons when dried and yield 1 ton of ash from which not more than 25 to 30 lb., more often scarcely half this quantity, of iodine is obtained. Potash salts constitute a joint product at most plants and common salt also may be recovered. Alginate, a sticky, nitrogenous substance used for sizing or waterproofing textiles, is sometimes recovered from the raw weed.

For treating California oil-well brines, which contain only 30 to 70 parts of iodine per million, there are two successful processes. In one, the iodine is liberated in the acidulated brines by an oxidant, usually  $\text{Cl}_2$ , and absorbed in activated carbon; in the other, or silver process, it is precipitated quantitatively by adding  $\text{AgNO}_3$ . These waters contain considerably more  $\text{Br}_2$  than  $\text{I}_2$ , but recovery of  $\text{Br}_2$  has not been considered worth while.

*Markets and Prices.*—Chilean crude iodine is packed in wooden kegs holding about 60 kg., which are sewed in rawhide to protect them in shipping and also to hold together the staves when, as not infrequently happens, the steel hoops corrode and drop off after years in storage. The

Iodine Association specifies a minimum purity of 99 per cent and not more than 0.1 per cent acidity (calculated as  $H_2SO_4$ ), but even the crude product ordinarily runs 99.5 per cent. Iodine sublimes readily (b.p.  $184^\circ C.$ ) and is obtained in high purity by this treatment. Crude iodine has relatively few purchasers, principally manufacturers of its salts, many of which are official in the United States Pharmacopoeia. A rough estimate shows that it is eventually marketed about as follows: potassium iodide, 70 per cent; sodium iodide, 5 per cent; resublimed iodine, 15 per cent; all other compounds, 10 per cent.

Lack of iodine in the human system is said to be one cause of goiter, and iodine salts are employed for internal antisepsis, for treatment of high blood pressure, arteriosclerosis, and a variety of other ills of man and beast. In certain localities all table salt has to be "iodized," and some municipal authorities require iodide to be dissolved in the drinking-water supply for the community. Iodine and its compounds are valuable laboratory reagents and have important uses in photography. Uses are found also in dye manufacture, tanning, and other industries, but iodine is far too expensive in comparison with chlorine or even bromine to encourage its wide employment industrially. At present, the most promising field for expansion of consumption is the addition of potassium iodide to stock feed. This costs only 5¢ per ton of feed and is said to be of great benefit to animals. By extending the medicinal uses of iodine to cover animals as well as humans, a fivefold expansion in consumption is envisioned.

Selling prices of crude iodine were fixed for many years by the Chilean Producers' Association at 12d. an ounce, or about \$3.89 a pound, f.a.s. vessel, with a 2.5 per cent discount on 500-lb. orders. Delivered at New York, it was worth around \$4 a pound. It has been estimated that the nitrate companies could have sold iodine profitably at 25¢ a pound but so long as the only competition was from seaweed iodine, costing \$2 to \$3 a pound, it was naturally more profitable to peg the price by international agreement. As development of an American industry, after 1931, pointed to a probable cost for iodine from oil-field waters of around \$1 a pound, the Chilean group (Corporation de Ventas) withdrew from the international cartel and sought to reestablish themselves in the important American market. Prices were progressively reduced and efforts were made to expand demand through laboratory research and a general educational program.

By May 1, 1936, the New York price of crude iodine was cut to 90¢, resublimed iodine was down to \$1.65 to \$1.50 a pound, and leading salts were correspondingly reduced. These prices were subject to a discount of 5 per cent applicable to purchases aggregating 2000 lb. during a 6-months period. On Oct. 10, 1936, the price of crude iodine was once more revised downward to 81¢ a pound with no quantity discounts. This is the

world price quotation, other currencies being referred back to American exchange.

### MEERSCHAUM

Meerschaum ( $H_4Mg_2Si_3O_{10}$ ) is a soft (hardness, 2 to 2.5), fine-grained, earthy, white, gray or yellow material. The name "meerschaum" (German for "sea foam") suggests its light weight; the specific gravity is given as 1 to 2, but when pure and dry it will float on water. Owing to its resemblance to the porous, bonelike structure of the cuttlefish or sepia, the mineral is also known as "sepiolite." When wet the material is soft, even somewhat plastic, but it becomes hard and tough after drying again, the toughness being more apparent in varieties that have a fibrous or leathery texture.

There are a few scattered deposits of meerschaum in the United States that have yielded a small production, but the world's supply of the material is obtained principally from Asia Minor. Meerschaum deposits near Eskishehir, Turkey, have been worked for centuries, possibly as early as 2000 years ago, and during the last 100 years their product enjoyed a large European market, being much carved in Vienna. World production, virtually all from Turkey, is reported to have exceeded 10,000 boxes, weighing 30 to 35 kg. each, in 1869. According to some reports, the exports in prewar years often amounted to 7000 boxes annually. Since about 1924, however, the output has declined, the annual average being scarcely 500 boxes. American deposits may have yielded a total of 1000 tons, chiefly from a deposit near Sapillo Creek, N. M. Meerschaum has also been obtained from the islands of Euboea and Samos, Greece; near Hrubshitz, Moravia, Czechoslovakia; in Bosnia; in Morocco; and near Vallecas, Madrid, and Toledo, Spain; but mines in those countries, never extensive, were closed many years ago. A related mineral, aphrodite, is found near Langbanshyttan in Vermeland, Sweden.

*Mining and Preparation.*—Meerschaum usually occurs as an alteration product of magnesite, serpentine, or possibly impure opal high in magnesium. The Turkish deposits occur in a valley filled with drift material from the surrounding mountains. The drift has been partly cemented by lime and the meerschaum occurs as scattered nodules, the size ranging from that of an egg to that of a football. These nodules are brought to the surface and worked on the spot, those from the dry pits being worth more than those from the wet. They are sorted according to complex grading, depending upon size and quality, before packing in the boxes. The mining is primitive, pits are dug by pick and shovel to a depth of 60 to 100 ft. with only enough timbering to allow the workmen to climb up and down. Nodules are recovered by a system of

untimbered drifts and crosscuts, the ore being hoisted to the surface in baskets by a windlass. Floods and cave-ins are common occurrences. Government aid, largely in the form of loans to the owners of the pits, has been extended recently to the Turkish mining industry but, according to latest reports, the Eti-Bank had not taken over the working of the pits.

*Uses.*—Virtually the only use of meerschaum is in smokers' articles, and meerschaum from any source but Turkey seems to have proved unsuitable. The making of meerschaum pipes was begun more than 200 years ago (1723) in Budapest. Somewhat later, in 1767, the industry was undertaken in Ruhla in the Thuringian Forest, Germany, and subsequently the manufacture of pipe bowls and cigar holders from genuine and artificial meerschaum has been centered principally in that town. The making of meerschaum pipes consists in roughly cutting the irregular masses to shape, carving by hand or turning down on lathes, and smoothing with glass paper and Dutch rushes. They are then boiled in wax, stearin, or spermaceti and polished with chalk or bone ash. The color of meerschaum deepens with smoking, owing to absorption of a mixture of nicotine and the finishing wax. The color continues to deepen as long as any wax remains, but when the desired color is reached it may be set by removing the remaining wax and boiling in linseed oil to fill the pores and harden the mineral. Details of the color-setting process are trade secrets. Before setting the color may be destroyed if the pipe is subjected to excessive external heat (such as washing in very hot water or drying too quickly). Artificial meerschaum pipes may be made from meerschaum chips and dust compressed into blocks. Imitation meerschaum is prepared by treating hardened plaster of Paris with wax and coloring with gamboge or other pigments. Pipes made from this material are not susceptible to color change, as are the genuine meerschaum pipes.

At Vallecas, Spain, meerschaum is said to be used as a light building material, and in Turkey and Algeria it has been employed in place of soap. Meerschaum has also been used as an ingredient in porcelain.

*Markets and Prices.*—Aside from the sporadic domestic production, much of which was unsalable, all meerschaum used in the United States has been imported. Imports amounted to 16,646 lb. valued at \$22,649 in 1924, but dropped to 788 lb. valued at \$2,536 in 1933.

Market quotations relate to cases of standard size and (like those for oranges) vary according to the size of the individual pieces in the container. The number of pieces per case may range from only 35 to several thousand. For material large enough to be made into pipe bowls the size is not so important as the quality, and for each of these sizes there are as many as seven grades ranging in price from \$155 to \$335 a case. Small pieces sell as low as \$30 a case. It is difficult to translate these quotations to a weight basis but the statistics for imports into the

United States show that the average foreign market prices per pound have ranged from a minimum of \$1.60 to well over \$3 per pound in recent years.

Under the Tariff Act of 1930 (paragraph 1552) a duty of 20 per cent ad valorem is levied on crude meerschaum.

#### QUARTZ\*

Silica ( $\text{SiO}_2$ ) or quartz occurs in crystalline (quartz, tridymite, and cristobalite), cryptocrystalline (chalcedony) or amorphous (opal, diatomite) forms. As generally used, the term "quartz" includes practically all sands, sandstones and quartzite, as well as rock or vein quartz. Next to the feldspar group quartz is the most abundant mineral in the earth's crust; and it appears in a greater number of varieties and modes of occurrence. Silica, free or in combination with other elements, constitutes 59.08 per cent of the lithosphere, and quartz alone represents fully 12 per cent of all rocks (ref. 26 at end of chapter). Being hard (No. 7 on Mohs' scale), moderately tough, resistant to acids, refractory, and often transparent, quartz has physical and chemical properties which, particularly in view of its abundance in nature, have led to a multiplicity of uses in industry.

Production statistics for quartz include the statistics for sand and gravel, sandstone, quartzite, and many other items that are separately discussed in this volume. Quartz crystals are mainly imported from Brazil and are not produced in the United States; that is, if we except the rather small tonnage of crystalline quartz saved out by certain feldspar miners and eventually marketed as ground quartz for ceramic uses. In 1935, the production of quartz in the United States from pegmatites, veins or quartzite amounted to 17,178 short tons valued at \$111,784, of which 7586 tons of an average value of \$3.53 a ton was marketed (mostly to grinding mills) crude or roughly crushed and 9592 tons valued at \$8.87 was ground by the original producers.

Quartz rock and sand may be priced at less than 50¢ a ton, and a good deal of lump quartz is available in certain localities at only \$1 or \$2 a ton, or about the same price as glass sand. Pulverized silica competes in a slightly lower price range with tripoli and other "soft silicas" varying all the way from about \$6 to \$35 a ton, the latter price being for a high-quality air-floated grade in carload lots, and higher prices being asked for smaller quantities. At the top of the list is rock crystal, which is nominally around \$2 a pound, or \$6000 a ton.

#### NONFUEL GASES

Natural gas, in ordinary parlance, is an aggregate of hydrocarbons, chiefly methane, but the gas from certain fields contains from a trace to 2 per cent or more of helium, together with some argon and neon;

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\* See also chapters on Abrasives, Dimension Stone, Granules and Special Sands.

nitrogen and carbon dioxide are even commoner components, some gases carrying over 80 per cent  $N_2$ . Hydrogen sulphide occasionally is present in objectionable amounts, excessively "sour" gases requiring processing to remove it. A few gas wells produce highly concentrated carbon dioxide and gaseous or vaporous emanations almost universally accompany volcanic activity. Of the volcanic gases, water vapor is naturally the most abundant and has been harnessed to steam turbines for commercial power production, notably in Italy where boric acid is jointly recovered from the vapor springs. Dry fumaroles emit sublimates of metallic salts, chiefly chlorides, mixed with atmospheric air. Acid fumaroles pour out water vapor charged with hydrochloric acid with minor amounts of sulphur dioxide, whereas in other fumaroles the water vapor is heavily charged with hydrogen sulphide or free sulphur, the latter being sometimes recovered commercially. Sulphur dioxide and carbon dioxide, says Clarke (ref. 27, at end of chapter), never occur together, although hydrogen sulphide and carbon dioxide often come from the same fumarole, the proportion of carbon dioxide gaining as volcanic activity diminishes. The gases that issue from an active crater contain superheated steam, hydrogen, carbon monoxide, methane, vaporized metallic chlorides, and a variety of substances of minor importance such as oxygen, nitrogen, argon, sulphur, and fluorine compounds. As temperatures drop, the nature of the exhalations changes until finally only steam, with possibly a little carbon dioxide, remains. Aside from fuel gases, steam, boron compounds and sulphur (certain deposits of which are characteristically of fumarole or solfataric origin), the commercial natural gases that are taken from the earth are helium and carbon dioxide.

*Air.*—The air we breathe, however, can be described as a mixture of gaseous minerals, and "mining air" yields compressed air for power transmission and other purposes, and liquid air. Liquid air has been used as an explosive, but principally as a source of oxygen. Oxygen, 99.5 per cent pure, is isolated in enormous quantities for use mostly in welding, though substantial quantities are used in treating pneumonia and other respiratory diseases. Nitrogen from the air has broken the Chilean nitrate monopoly, thereby making explosives manufacturers in leading countries independent of overseas deliveries of raw material and giving the farmer an unlimited supply of cheap fertilizer. Argon and neon likewise are won from the air, the former being used to fill ordinary incandescent electric lamps and the latter for the now familiar illuminated tubes for advertising signs as well as in sodium vapor lamps, glow lamps, and sundry electrical "trouble-shooting" devices. Krypton and xenon, the remaining "minerals" of the air, are far too scarce to command commercial significance; they can now be purchased, but a quantity sufficient even to inflate a football would cost hundreds of dollars.

*Helium* is colorless, absolutely inert—odorless, tasteless, nonpoisonous, nonflammable. As a lifting medium for balloons and airships, its principal use, it has a lift of 66 lb. per 1000 cu. ft. compared with 71 lb. for hydrogen, the lightest known gas. The United States Bureau of Mines is chiefly responsible for making helium available to the Army and Navy of this country in quantities of millions of cubic feet a month at a cost of less than one cent per cubic foot. As recently as 1917, it sold for \$2500 a cubic foot and in very small quantities. Construction of the first large-scale plant was undertaken at Fort Worth before the Armistice but this plant was closed in 1929 and the needs of the military arms of the United States now are met by a plant at Amarillo, Tex., constructed and operated by the Bureau of Mines, which makes 90 per cent recovery and a 98 per cent pure product. The method of recovery is to liquefy all the components of the gas except the helium. Operating costs vary from \$7 to \$12 per 1000 ft. according to the volume produced in a given year, being naturally much less when the plant capacity is more fully utilized. The residue gas is sold to a local gas company for fuel purposes. Two small, privately owned helium plants have been operated intermittently at Dexter, Kans., and Thatcher, Colo. The production of helium is closely geared to the operation of large lighter-than-air craft. Other uses, of minor importance, are for filling small toy balloons, as an inert atmosphere for certain metallurgical operations, as a safeguard against the "bends" in deep-sea diving, in radio tubes and electric signs, and for cooling electrical equipment. Significant, perhaps, is its use in the sick room, mixed with oxygen for treating pulmonary diseases.

The only known sources of low-cost helium are a few natural-gas fields in the United States, where gas containing upwards of one per cent of helium has been found not only in the Texas Panhandle but also in southeastern Kansas, Ohio, southeastern Colorado, and eastern Utah. Gas containing as much as one per cent helium has not been found in any other country except Ontario, Canada, and there the supply is too small and rock pressures are too low for commercial production. Domestic supplies occur in formations ranging in geologic age from Cambrian to Tertiary. The fact that the reservoirs so often lie close to crystalline rocks, however, has been interpreted as indicating that the helium may have been derived from the breakdown of radioactive elements presumably present in minerals such as pitchblende and monazite in the basement rocks. In England, helium has been made from Indian monazite, but this source would seem to be far too expensive; a ton of monazite, costing not far from \$60, yields only 25 cu. ft. of the gas. The atmosphere, too, contains helium but the proportion of helium present is so small—only one part in 185,000—that it cannot be recovered economically except as a byproduct and probably never in quantities



that would suffice for an air navigation program of any real magnitude.

*Carbon Dioxide.*—At ordinary temperature carbon dioxide is a colorless, odorless, noncombustible gas about  $1\frac{1}{2}$  times as heavy as air. It dissolves readily in water and can itself be liquefied by compressing to 650 lb. per sq. in. Solid carbon dioxide (dry ice, Dioxide, etc.), although it has the appearance of packed snow, weighs about  $1\frac{1}{2}$  times as much as water ice, has a temperature at ordinary atmospheric pressure of  $-109^{\circ}$  F. ( $-78.5^{\circ}$  C.) or lower, and evaporates without becoming liquid. Only when the pressure exceeds about five atmospheres and the temperature is above  $-70^{\circ}$  F. (the "triple point" pressure and temperature) can carbon dioxide exist as a liquid.

Until about 1926 carbon dioxide was obtained chiefly from gases of coke ovens, lime kilns, metallurgical plants, fermentation plants and chemical plants, but more recently increasing quantities have been produced from natural gases. In the United States gas wells suitable for producing solid carbon dioxide are principally in Colorado, Utah and New Mexico, although California also has been a large producer from this source. The gas occurs, as does helium, in rocks of Cambrian to Tertiary age; the high carbon dioxide content may be derived from oxidation of hydrocarbons with mineralized waters or the reaction of hot magmas on limestone. Mexico is one of the leading foreign producers because of the quality and persistence of the supply in the Pánuco field, near Tampico. Canada, France, Germany, Italy, India, New Zealand, Japan, and probably other countries have utilized natural sources. Gas-bearing springs have been a source of carbon dioxide in several states, notably at Saratoga Springs, N. Y., and Manitou, Colo. Plants for making solid carbon dioxide have recently been built in all leading countries of Europe, Japan, British India, Australia, and several Latin American countries.

Production of liquid carbon dioxide increased in the United States from 23,978 tons valued at \$2,345,743 in 1909 to 44,093 tons valued at \$6,280,647 in 1929. Solid carbon dioxide was scarcely produced at all prior to about 1925 and was commercially unimportant prior to 1929, when production jumped to around 15,000 tons. In 1933, the Census reported 58,431 tons of liquid, of which 32,250 tons was used in making solid; the production of solid was reported as 29,789 tons valued at \$1,972,130 for that year; subsequently it has more than doubled.

There are several methods for making solid carbon dioxide from the purified gas, but in most of them the liquid is partly converted to snow in an expansion chamber and the snow is then compacted in the same chamber by repressing.

Liquefied gas was formerly used almost exclusively for carbonating beverages, but now its principal use is for making solid carbon dioxide,

which is consumed mainly by makers of ice cream and in shipping perishable goods by truck or train. Minor uses for the solid are found for quick cooling in various industries—in making golf balls, oil refining, candy making, and the manufacture of varnish and sundry chemicals. It may be used to stop the flow of water when making repairs to pipes and conduits, to freeze quicksands in shaft and tunnel driving, and to kill rats and vermin. Liquefied carbon dioxide (Cardox) is increasingly used in mining as a safe, slow-acting explosive. The gas has been employed as a refrigerant in mechanical refrigerating machines but seems less satisfactory for this purpose than ammonia, sulphur dioxide, Freon ( $\text{CCl}_2\text{F}_2$ ), or methyl chloride.

Carbon dioxide in cylinders was quoted in October 1936 at 6¢ to 8¢ per pound at New York. A frequently quoted price for solid carbon dioxide is \$37.50 a ton, the range for wholesale quantities in different parts of the United States being from about \$30 to \$50 a ton.

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## CHAPTER XXX

### MONAZITE

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**M**ONAZITE is one of the "has beens" among minerals. Following the era of kerosene lamps, when gas was the best and most convenient illuminant, monazite, through its child, the incandescent gas mantle, was a boon to civilization in saving eyestrain through better illumination of town and city dwellings and streets. Almost every new industry rises by the wreckage of older ones, and when improved electric lamps supplanted the incandescent gas mantle, tungsten became the new favorite and monazite has been all but forgotten. A small business yet remains—whether some new discovery or development will bring back demand for the mineral no one knows.

#### COMPOSITION

Monazite is not well understood. It is easily stated to be a monoclinic phosphate of the cerium metals, essentially  $(\text{CeLaDi})\text{PO}_4$  or  $(\text{CeLaDi})_2\text{O}_3 \cdot \text{P}_2\text{O}_5$ , but nearly all specimens contain thorium and silica, usually in quantities suggesting thorium silicate, though in some the percentages are far from such a ratio. The thorium content ranges from 0 to about 18 per cent in published analyses. Monazite as it commonly is found, in tiny grains, is a very different mineral in appearance from the large crystals or masses that are found in pegmatite. The grains are honey yellow or in tints of brown to reddish and are resinous subtranslucent to subtransparent. The large crystals are commonly dull red and opaque. Monazite has one perfect cleavage (parting?) *c*, distinct on *a*, difficult on *b*; fracture, conchoidal to uneven; brittle; hardness, 5 to 5.5; sp. gr., 4.9 to 5.3, mostly 5.0 to 5.2.

Although there are many other thorium-bearing minerals, none is plentiful or cheap enough to compete with monazite.

#### GEOLOGY AND DISTRIBUTION OF DEPOSITS

The geology of monazite is simple. It is found in bright, resinous grains, mostly less than one millimeter in diameter, in gneisses that have been soaked in pegmatitic solutions. In the pegmatites themselves monazite may develop as large crystals or masses. A single crystal has been known to reach a weight of 30 lb. No primary deposits in either the

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gneisses or the pegmatites, rich enough to pay for exploitation, have yet been discovered. Monazite is extremely resistant to weathering, and as its mode of occurrence is very much the same as that of zircon and ilmenite the three minerals are found together in some black sands.

*United States.*—In the United States the principal monazite deposits have been found in the Piedmont region of the Carolinas, extending about 150 miles northeast from northeastern Georgia. Here the Carolina gneiss is the host rock. As a result of weathering and erosion the monazite has collected in the stream gravels of the region. Very little rich gravel was ever found, there were no very large bodies of gravel, and about 15¢ a pound seemed to be the minimum price at which the monazite could be produced profitably. Not only was there little rich gravel but the thorium content of the monazite was comparatively low. In 10 monazite concentrates noted by Pratt and Sterrett<sup>13</sup> the thorium content ranged from 1.27 to 6.54; only three had 6 per cent or more, and five were below 2.5 per cent. In the paragraph preceding the list of analyses they say that the thorium content ranged "from 0.01 to more than 7 per cent." The average content of the Carolina monazite was less than 6 per cent  $\text{ThO}_2$ . No other part of the United States has produced more than a very few tons of monazite, although efforts have been made to work stream sands near Centerville, Idaho, and to separate monazite from the black sands of Pablo Beach (now Jacksonville Beach), Florida, which carried about 0.09 per cent of monazite.

*Brazil.*—The first large beach deposits of monazite were found in Brazil on the coasts of Espirito Santo and Bahia, in 1895, and soon made operation difficult on the leaner deposits of the United States. The deposits lie for long distances along the coast, on raised beaches behind the present shore, and along the rivers flowing into the Atlantic through the raised beaches. The monazite carries about 6 per cent  $\text{ThO}_2$  and 0.15 to 0.25 per cent  $\text{UO}_2$ . The monazite is accompanied by ilmenite and zircon, which have been shipped in large quantities. There are also smaller quantities of numerous other minerals.

The sands are first concentrated by sluicing and, after drying, the concentrates are separated by electromagnetic machines.

Up to 1927, Brazil had recorded a production of 70,693 short tons (of 2000 lb.) and possibly 1000 tons has been exported since that time.

There is undoubtedly much more monazite to be obtained at the prices that formerly prevailed, about \$100 per ton. A low of \$43 was recorded in 1898, and a high of \$141 in 1912.

*India.*—In 1911, about the time that monazite was at its highest price and, ironically, at a time when the tungsten lamp was already undermining the market for monazite, the State of Travancore, on the

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<sup>13</sup> References are at the end of the chapter.

west side of the southern point of India, began to supply the market with amber colored monazite in particles 0.1 to 0.2 mm. in diameter carrying between 8.8 and 10.08 per cent  $\text{ThO}_2$ —from one-half to two-thirds more than the Brazilian monazite.<sup>4</sup> According to official reports, nearly 18,000 short tons was exported from 1911 to 1933 inclusive. In 1933, the price per ton for 156 short tons was \$51. No later figures are at hand. The principal monazite beaches are the same as the ilmenite and zircon beaches, those of Manavalakurichi and Quilon. Other deposits lie east of Cape Comorin in the Tinnevely district and near Waltair in Vizagapatam.

*Other Monazite Beaches.*—Ceylon has beaches carrying considerable monazite and the beach that stretches along the northern coast of New South Wales and southern Queensland carries some monazite that can be saved as the sands are worked for rutile, ilmenite and zircon.

#### POLITICAL AND COMMERCIAL CONTROL

Great Britain controls the richer deposits of monazite, but Brazil apparently has larger deposits in which the mineral carries only about two-thirds as much thorium. Germans probably control the Brazilian deposits commercially. The manufacture of thorium nitrate is divided between German, British, French and American firms, the first named being probably the largest manufacturers.

#### TESTS

The indices of refraction of monazite are high, ranging from 1.786 to 1.849, so they are out of reach of ordinary liquids for microscopic determination and the piperine-iodide melts must be used, but the height of the indices is one qualitative factor. For testing Fermor<sup>4</sup> recommends a Browning direct-vision spectroscope. This shows the didymium lines.

Monazite sand can be easily cracked between the teeth; a test for phosphorus, a test for radioactivity by an electroscope, and specific gravity determinations are confirmatory tests. Though no single test is conclusive, yet when made on minerals that have proper color and appearance and that are from favorable localities, the tests given afford a fair degree of assurance.

#### USES

Thorium to be made into nitrate for the manufacture of incandescent mantles to be used with gas, gasoline or kerosene still is the component of monazite in most demand. Although India and China are the two best customers, 2 tons of thorium nitrate is consumed annually in Sweden,\*

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\* C. E. Dickerson, Jr., U. S. Com. Attaché, Stockholm. *World Trade Notes*, Chem. and Allied Prod. (1936) 10, 6.

and some is still used in the British Isles and other northern European countries. Some mantles are used in light and heat therapy.

The mesothorium, which invariably accompanies the thorium, is saved to be used as a substitute for radium, and the mixed residue of salts of cerium, didymium, lanthanum, etc., is reduced as an alloy of iron and used as a spark producer in lighters for cigarettes, carbide lamps, etc. The reduced alloy of the residual rare-earth metals known as "misch metal" has been used also in a small way in steelmaking and in cast iron. Many uses for the metals in glass and in chemicals have been proposed, but they actually consume inconsequential quantities. Some cerium is used as a mold preventive in cloth. For the purpose it is introduced as nitrate, and the cloth is heated to drive off the nitrate radical. A little cerium has been used in flaming arc lamps.

Monazite is moribund. Should it revive there are large supplies available at prices comparable to those of the past.

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## CHAPTER XXXI

### NATIVE BITUMENS

By A. H. REDFIELD\*

**B**ITUMENS have been defined by Abraham<sup>1</sup> as substances of variable color, hardness, and volatility, composed principally of saturated hydrocarbons, substantially free from oxygenated bodies, sometimes associated with mineral matter, the nonmineral constituents being fusible and largely soluble in carbon disulphide. The principal bitumi-

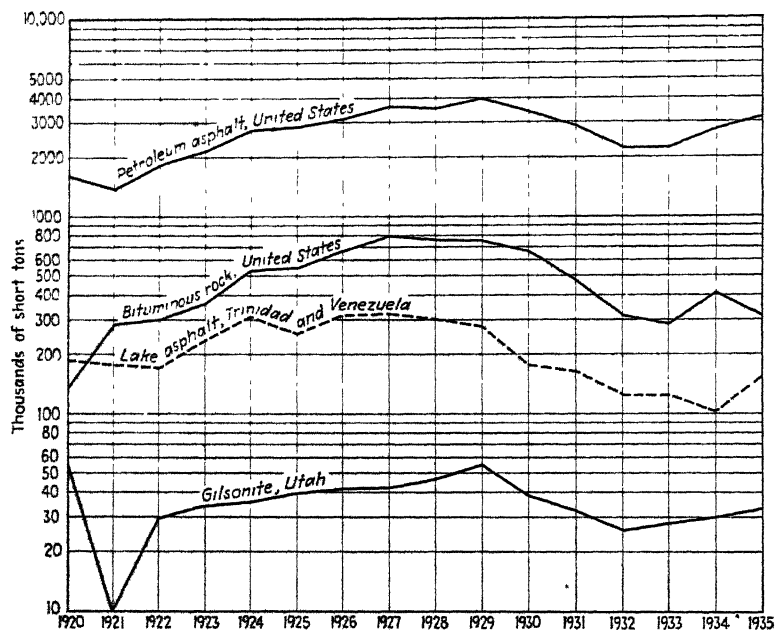


FIG. 1.—RATE OF GROWTH OF SALES OF PRINCIPAL BITUMENS BY PRODUCERS, 1920–1935.

nous substances (other than petroleums) known to commerce are native asphaltites, such as gilsonite, grahamite and glance pitch, distinguished by their hardness and relatively high fusing point; native asphaltic pyrobitumens, such as wurtzilite, distinguished by their infusibility and comparative insolubility in carbon disulphide; mineral waxes, such as ozokerite, distinguished by their high content of crystallizable paraffins;

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<sup>1</sup> References are at the end of the chapter.



and native asphalts, containing varying amounts of mineral matter, distinguished by the comparative absence of crystallizable paraffins; as well as bituminous residues obtained from the distillation of petroleum.

*Gilsonite*.—Gilsonite, a native asphaltite 98 to 99.9 per cent pure, characterized by a hardness of 2 (Mohs), a specific gravity of 1.01 to 1.10, and a melting point of 230° to 400° F. (K. and S.), is mined commercially only in northeastern Utah. It occurs in almost vertical veins from a few inches to 18 ft. wide and 8 to 30 miles long that cut across the Tertiary limestone and shale of the broad Uinta Basin. The bitumen is believed to have been distilled by heat from oily material in the underlying Green River shale.

At the Rainbow mine of the Gilson Asphaltum Co. (General Asphalt) the gilsonite, formerly extracted in an open cut by a system of slopes and benches, is now mined by shafts and horizontal drifts. Little timbering is required. The mining methods of the Raven Mining Co., of Chicago, and of the American Asphalt Association and the Utah Gilsonite Co., both of St. Louis, are equally simple. To avoid dust explosions and shattering of ore, explosives are little used. The brittle gilsonite is dug out with picks, hand-selected, sacked, and hoisted to the surface.

"Select gilsonite" and "jet asphaltum" are used principally in the manufacture of varnishes and japans, printing and rotogravure inks. "Standard gilsonite" is employed in the manufacture of storage-battery cases and molded articles, brake linings, tile and mastic, sealing compounds, insulation, and wood stains. Weathered gilsonite from the surface is used to saturate roofing felt and for blending with rubber.

Sales of gilsonite by producers reached a peak of 54,987 short tons in 1929, declined to 25,955 tons in 1932, and rose to 33,227 tons in 1935. Since 1925 the value, f.o.b. mine or shipping point, has ranged from \$19.43 to \$22.90 per ton, averaging \$20.91.

*Wurtzilite*.—Wurtzilite, commercially called "elaterite," is an asphaltic pyrobitumen characterized by a hardness of 2 to 3, by a specific gravity of 1.05 to 1.07, and by its infusibility. It occurs in narrow veins, 1 to 22 in. wide, 100 ft. to 3 miles long, that cut the Tertiary beds of the Uinta Basin of northeastern Utah. It is mined 50 miles southwest of Fort Duchesne, Utah, by the Raven Mining Co. of Utah, Chicago, Ill., by horizontal drifts and stopes.

Crude wurtzilite, an insoluble, refractory mixture of hydrocarbons, is heated under pressure to 500° to 580° F. The vapors evolved are condensed and returned to the still, where they reduce the material to a plastic mass, which, after heating, is converted to a substance soluble in carbon disulphide and moderately soluble in 88° naphtha, known as "Kapak," or wurtzilite asphalt. Wurtzilite asphalt is used in the manufacture of paints and coatings for weatherproofing and preventing corrosion, for electrical insulation, and for blending with rubber. From a

maximum of 280 short tons in 1927 sales of wurtzilite declined to 25 tons in 1932 and rose to 61 tons in 1935. The average sales value at the mine has declined from \$90 a short ton in 1931 to \$79 in 1935.

*Grahamite and Glance Pitch.*—Grahamite, an asphaltite with a specific gravity of 1.15 to 1.20 and a fusing point of 350° to 600° F. (K. and S.), was mined in Pushmataha County, Okla., until 1924 and formerly in Ritchie County, W. Va.

In Cuba veins of grahamite and glance pitch, cutting a complex of sedimentary and serpentinous rocks, have been mined by American, British and Cuban companies. At Mariel, in Pinar del Rio, nearly vertical veins of grahamite 6 to 30 ft. wide and 300 ft. long are mined by horizontal crosscuts and drifts and stoped out by manual labor. The crude material, 55 to 58 per cent pure, with a specific gravity of 1.49 and a melting point of 374° to 400° F., is picked, hoisted and sacked either in crude form or after steam refining. At Talaren, in Camaguey, a steep vein of glance pitch with a specific gravity of 1.12, a hardness of 2, and a fusing point of 315° to 345° F. (K. and S.), has been mined by shafts, drifts, and stopes. Exports of grahamite and glance pitch from Cuba, chiefly to the the United States and the United Kingdom, amounted to 15,040 short tons in 1931 and 8131 tons in 1935.

*Ozokerite.*—Ozokerite, a native wax mined in Polish Galicia and formerly near Soldier Summit, Utah, is believed to have been derived from the polymerization of paraffin-base petroleum. It is used principally for the manufacture of wax candles and petrolatum, for electrical insulation, and for waterproofing. In Galicia, along the overthrust front of the Carpathians, rich veins of ozokerite that fill veins in fractured Oligocene and Miocene beds are mined by shafts and galleries in the districts of Drohobycz and Stanislawow. From 994 short tons in 1930 the production of ozokerite in Poland declined to 396 tons in 1935. Most of the output is exported to Germany for refining.

*Lake Asphalt.*—In southwestern Trinidad, at Brighton, a lake of natural asphalt that covers approximately 114 acres and is nearly 300 ft. deep at the center has been worked for years by subsidiaries of the General Asphalt Co. of Philadelphia. This emulsion of bitumen, water, mineral, and vegetable matter is believed by Richardson<sup>20</sup> to be derived from heavy asphaltic oil in the underlying Miocene in contact with colloidal clay and silica. Crude asphalt, which contains 33 per cent of water and 27 per cent of mineral matter, is dug by manual labor from the surface of this lake, loaded into cars, and hauled by cable to the refinery, where it is dehydrated in open tanks and heated by steam. Some crude asphalt is transported in bulk cargoes to the United States, where it is refined for domestic uses.

Refined Trinidad asphalt has a specific gravity of 1.4, a hardness of 1 to 2, and a softening point of 183° to 189° F. (K. and S.). It contains

54 to 56 per cent of bitumen 38.5 per cent of colloidal clay and silica, and 4 per cent of water of hydration. It is used for paving and for the manufacture of roofing, waterproofing, mastic, molding compounds, pipe coatings, insulation and paints. Production of lake asphalt in Trinidad, which averaged about 222,000 short tons a year from 1920 to 1929, amounted to 150,727 tons in 1935.

In eastern Venezuela, a subsidiary of the General Asphalt Co. formerly worked the Bermudez lake of asphalt, near Guanoco.

From 77,042 short tons in 1924 the production of lake asphalt in Venezuela declined to 7421 tons in 1932, when production ceased.

*Bituminous Rock.*—In west central Kentucky extensive deposits of bituminous sandstone occur in Edmonson, Breckinridge, Grayson, and Hardin Counties in the Chester formation (upper Mississippian) and the Pottsville formation (Lower Pennsylvanian). The asphalt is believed by Russell<sup>21</sup> to be derived from petroleum, altered by the oxygen contained in circulating waters. At Kyrock, Edmonson County, the Kentucky Rock Asphalt Co. of Louisville mines the bituminous Pottsville sandstone throughout the year in large open pits. After being crushed, pulverized, mixed by machinery and delivered by conveyer belts to barges, the rock is floated by river to Bowling Green or Rockport. At Summit, Hardin County, the bituminous Chester sandstone is quarried by the Ohio Valley Rock Asphalt Co., of Louisville. It is crushed, pulverized, and loaded directly into open-top gondola cars, from which it may be unloaded by steam shovels or picks and shovels.

The prepared material, which contains 7 per cent of bitumen, may be laid cold to surface highways, railroad platforms, bridge floors and tennis courts. Nearly half of the rock asphalt is sold directly to states, counties and municipalities. From a maximum of 344,220 short tons in 1927, sales of Kentucky rock asphalt declined to considerably less than 100,000 tons in 1935. The average sales value per short ton declined from \$9.17 per short ton in 1927 to \$6.49 in 1935.

In northern Alabama, at Margerum, the Alabama Asphaltic Limestone Co., of Birmingham, mines a Middle Mississippian oolitic limestone in open quarries. The rock, which contains 2.5 to 9.5 per cent of bitumen is crushed and milled, fluxed with petroleum asphalt, and sold for paving purposes.

In Uvalde County, Texas, an Upper Cretaceous coquina limestone that contains 9 to 12 per cent of bitumen is drilled and blasted down in open pits by the Texas Rock Asphalt Co., the Uvalde Rock Asphalt Co., and White's Uvalde Mines, all of San Antonio; it is crushed, pulverized, blended with asphaltic flux oil, and shipped by rail in open-top cars. The prepared material, which averages 10 per cent of asphalt, may be laid cold to surface highways. Floor tiles and plastic planks are made with the extracted bitumen. From a peak of 376,770 short tons in 1927, sales of

Texas rock asphalt declined to less than 75,000 tons in 1935. The average sales value per short ton at the mines was \$3.33 in 1927 and \$3.24 in 1935.

In the Arbuckle Mountains of south central Oklahoma nearly horizontal beds of Ordovician bituminous sandstone and limestone are quarried along the face, crushed, mixed, and sold for paving purposes by the Southern Rock Asphalt Co. of Oklahoma City.

Rock asphalt was also produced in 1935 in California, Kansas, Missouri, and New Mexico.

*Competition with Petroleum Asphalt.*—In all of their industrial uses native bitumens and rock asphalt compete with asphalt manufactured from petroleum. To the advantages of large-scale production and extensive marketing organization enjoyed by petroleum asphalt, the native asphaltites have opposed their physical characteristics for special uses and bituminous rock its availability for immediate laying.

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## CHAPTER XXXII

### NITRATES

BY HORACE R. GRAHAM,\* MEMBER A.I.M.E.

**S**ODIUM nitrate, known in Chile as *caliche*, is variable in composition and may contain from 5 to 30 per cent each of nitrate, chloride and sulphate, all as sodium salts. Potassium, magnesium and calcium occur in percentages from 0.1 to 5, usually as double salts with sulphate which are but slightly soluble in nitrate leach solutions. These double salts are: bloedite ( $\text{Na}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 4\text{H}_2\text{O}$ ), glauberite ( $\text{Na}_2\text{SO}_4 \cdot \text{CaSO}_4$ ), polyhalite ( $\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 2\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). Some deposits contain up to 30 per cent of their nitrate content in the form of a double salt, Darapskite ( $\text{NaNO}_3 \cdot \text{Na}_2\text{SO}_4 \cdot \text{H}_2\text{O}$ ), which is insoluble in cold leach solutions but at elevated temperatures, 55° C. and above, is completely decomposed and soluble. Minor salts such as potassium perchlorate, sodium iodate and unknown borate materials exist in percentages from 0.02 to 1. The water-insoluble salts may vary from 20 to 80 per cent in the nitrate minerals.

The physical characteristics are variable, with color range from pure white to browns and reds, and occasional samples of yellow, black and blue. Nitrate mineral occurs in slimy, sandy or gravelly form, dependent upon the preponderance of the constituents that are cemented with salts to form a hard conglomerate.

#### ORIGIN, MODE OF OCCURRENCE AND DISTRIBUTION

No acceptable explanation of the primary origin of the nitrate minerals in Chile has been developed, but it seems definite that nitrate was finally collected in an inland sea, which eventually deposited part of its load along its shore lines. Finally the sea drained into the Pacific Ocean, but the deposits that had been laid down on the eastern shore were either dissolved or washed away by drainage from the Andes, or covered with alluvial debris, leaving the present deposits on the western shore of the inland sea or the eastern slope of the coast range.

The typical deposits occur on the comparatively low, level slopes on the east side of the coast range, and consist of a layer varying from a few inches to several feet. Above the nitrate mineral layer lies an overburden varying through the same ranges of thickness and composed of material from loose sand and dust to a firm conglomerate of rock fragments and salts.

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\* Vice-president, Lautaro Nitrate Co., Ltd.; Vice-president, Compañia Salitrera Anglo Chilena, Santiago, Chile.

The only known commercially exploitable deposits are those in the narrow strip between latitudes 19° and 26° South, roughly 450 miles long and from 10 to 50 miles wide, in the deserts of northern Chile. These deposits are extremely irregular in shape and of spotty location. A large proportion of the original nitrate has undoubtedly been leached from the ground by heavy rains, which occur about every 10 years, but several hundred million tons of recoverable nitrate still remain for exploitation. Small deposits similar chemically have been found in many parts of the world, in Africa, western Mexico, southern Peru, and the southwest of the United States, where similar arid desert conditions exist, but none are commercially exploitable.

#### POLITICAL AND COMMERCIAL CONTROL, PROSPECTING AND MINING

Effective July 1, 1933, there was created by law the Chilean Nitrate and Iodine Sales Corporation. This organization with five Fiscal Direc-

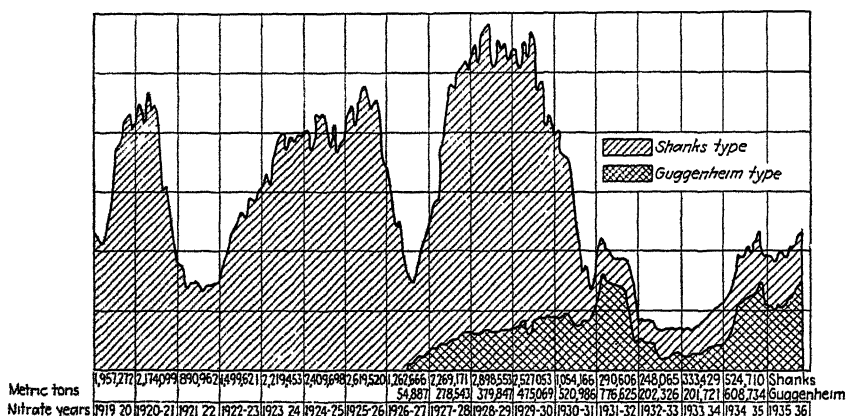


FIG. 1.—PRODUCTION OF CHILEAN NITRATE.

tors and five Industrial Directors, and a President, who by law must be Chilean, controls the commerce of nitrate and iodine. The producing industry is controlled by three large companies: The Lautaro Nitrate Company, Limited, the Compañía Salitrera de Tarapacá y Antofagasta and the Compañía Salitrera Anglo-Chilena, which represent 90 per cent of the entire industry, the remaining 10 per cent being in the hands of small independent producers. The Lautaro and Anglo-Chilena companies, representing over 60 per cent of the industry, are owned by American and British capital under American control—the Guggenheims—whereas the Compañía Salitrera de Tarapacá y Antofagasta represents British, German, American and Chilean capital with a predominating British influence. The independents are principally Chilean, Spanish, Yugoslav and French. The production and world consumption of Chilean nitrate since 1920 has been as shown on Figs. 1 and 2.

Previous to the World War, Chilean nitrate had a monopoly of the world's fertilizer market, and while some ammonium sulphate was being produced as a by-product in the coke industry in connection with the manufacture of steel, no really competitive synthetic processes were developed. However, Germany, consuming 800,000 tons of nitrate annually, was isolated during the war and forced to develop means of producing nitrate to save its political life. Technical experts and vast sums of money were available and as a result the Fritz Haber process, which had been partly developed before the war, was placed on a commercial basis. This process, which combines pure nitrogen and pure hydrogen under high pressure and temperature with the aid of a catalyst, is the mother patent for practically all synthetic processes—the Claude, Casale, Mont-Cenis and others.

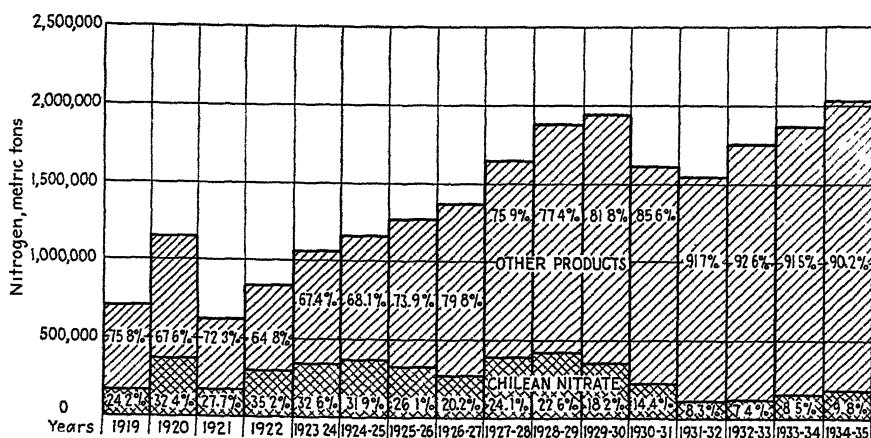


FIG. 2.—WORLD CONSUMPTION OF NITROGEN.

The successful development of the Haber process was followed by the construction of synthetic plants in Germany, Holland, Belgium, France, Italy, Great Britain, Norway, Czechoslovakia and the United States, and with the aid of national barriers and government subsidy these plants forced the Chilean industry into a subordinate position, where today it sells about 10 per cent of the world's nitrogen.

With the development of synthetic processes, cyanamid and many complicated chemical fertilizer products have become competitors.

Chilean natural nitrate contains vital impurities such as magnesium, iodine, boron, calcium, potassium, lithium and strontium, which in addition to the nitrogen content make an ideal product for the development of plant life and for use in connection with animal food.

Inasmuch as the *caliche* is usually found close to the surface, and in relatively small thicknesses, prospecting and exploration are rather simple. The present practice is to sink pits one meter in diameter in



promising localities, and cut a good sized sample channel vertically in each, sectionalizing the samples according to the variations in the ground so as to get as good a classification of overburden, ore and underlying material as possible. Pits are first sunk at wide intervals—perhaps 500 m. or even 1 km., and then at closer intervals when promising areas are located.

Both hand mining and mechanical mining are used. The former is used in the Shanks process plants, which demand a high-grade ore. In hand mining, the ore and overburden are blasted together, usually by hand-drilled holes, although sometimes air drills are used, then the

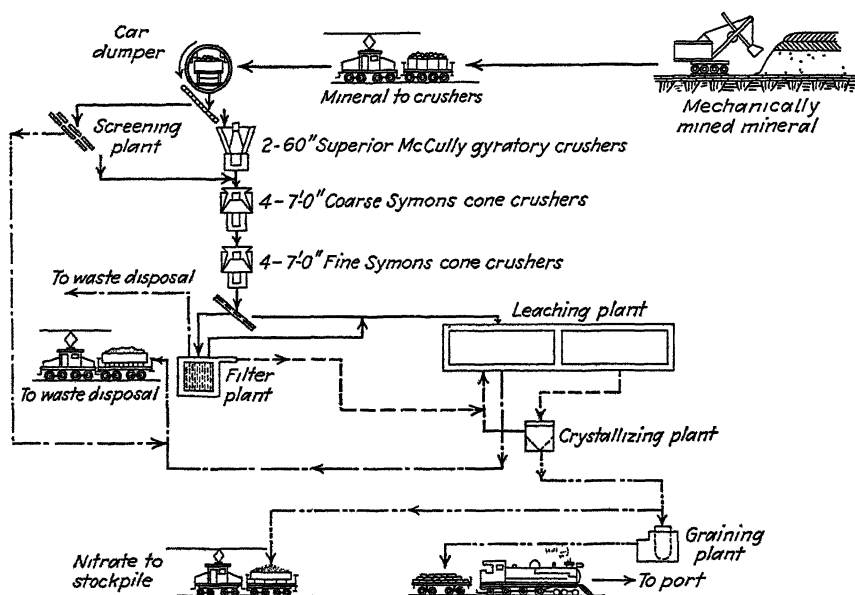


FIG. 3.—FLOWSHEET OF GUGGENHEIM MECHANICAL PLANT.

miner selects the ore and breaks it into pieces of convenient size, which he places upon a carefully squared up pile for measurement and hand-loading into cars for shipment to plant. A variant of this is underground mining of beds of high-grade *caliche* lying under overburden of thicknesses up to 10 or 12 m. Methods similar to hand mining are used, modified to suit conditions.

For the newer Guggenheim plants, the mechanical mining system is briefly as follows (Fig. 3): A loop system of electric haulage is laid out, in which main lines are electrified and shifting mining lines are not. Combination battery trolley 36-ton G. E. locomotives are used for ore haulage. Each mining section is equipped with a Bucyrus Erie 100-B electric shovel (Fig. 4) and a similar dragline. A mining section includes about 2000 m. of length between the empty line and the load line, and is usually laid out with a favorable grade for the ore trains, which operate

on storage battery through these sections. The dragline removes the overburden over a cut 14 m. wide, casting it into the previous cut. Part of the overburden has to be blasted. Ore is then drilled and blasted, and the shovel follows down the cut, loading into the ore cars. Track, airline and power-line towers are shifted as the cuts progress.

#### PREPARATION FOR THE MARKET

Sodium nitrate is now extracted from *caliche* in two types of plants. Until 1926 the only extraction plants in operation, of which about 150 were in existence, were known as the Shanks plants or "Oficinas."

In Shanks Oficinas, the *caliche* is brought from the mine, known as the *pampa*, in small cars up to 10 tons capacity, and crushed in jaw crushers to about 2½-in. maximum size and finally delivered to boiling

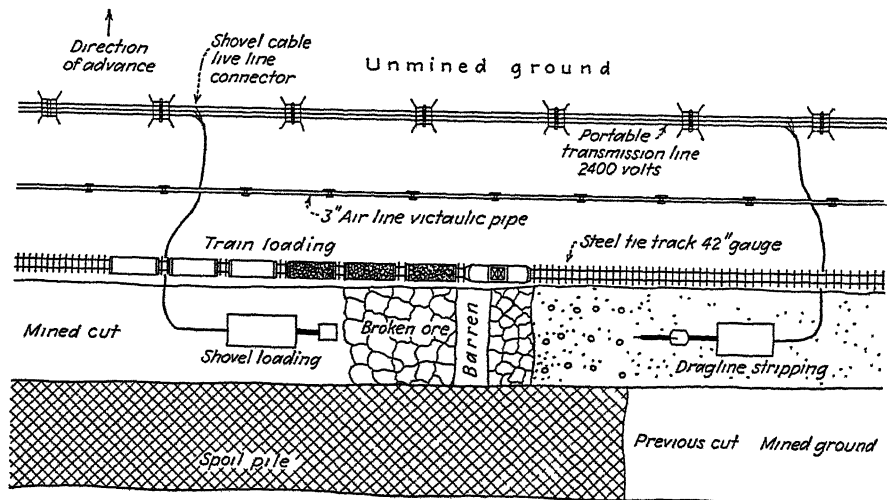


FIG. 4.—PLAN FOR MINING NITRATE ORES WITH POWER SHOVELS.

tanks of 50 to 90 tons capacity. In these tanks the *caliche* is leached at the boiling temperatures with a mother liquor containing about 450 grams per liter of nitrate. By contact with the *caliche* at the boiling temperature for 12 to 15 hr., the mother liquor dissolves more nitrate to bring the nitrate concentration up to about 700 grams per liter. The strong solution is then run to a settling tank to settle out the slimes, and finally the clear hot solution is run to large open tanks for cooling.

The residue in the boiling tanks is washed with a series of graduated washes and finally with water, and after draining is discharged by hand into small cars beneath the boiling tanks and hauled to waste. The residue contains from 3 to 8 per cent sodium nitrate, depending upon the slimy characteristics of the *caliche*.

The strong solution, after about 8 days cooling, is again reduced to mother liquor for use in a new cycle of operations. The crystal deposit

of nitrate, after the mother liquor is drained off, is shoveled into small cars and dumped onto a storage yard for further drainage, and in about three months is ready for bagging and shipping. The nitrate so produced is of 95.5 to 96 per cent purity, and contains 1 to 2 per cent moisture and small amounts of sodium chloride, sodium sulphate, borax, potassium perchlorate and iodine (as iodate), and other salts essential for the development of plant life.

The capacities of these Shanks Oficinas vary from 10,000 to 180,000 tons of sodium nitrate per year, and for their successful operation *caliches* of less than 13 per cent sodium nitrate cannot be treated economically.

In 1926 and 1930, two new large plants, known as Maria Elena and Pedro de Valdivia, were built embodying a new process—known as the Guggenheim process—each to produce over 500,000 tons annually. In this process the *caliche* is mined by power shovels and the mining recovery from the deposits is nearly 100 per cent of the nitrate content, but the average sodium nitrate content of the *caliche* is reduced to about 8 per cent. The new Guggenheim process is particularly designed for treating low-grade mineral.

The *caliche* ore is delivered in cars of 30 to 35 tons capacity and dumped by a revolving car dumper into the crushers. It varies in size from small fragments up to pieces of 2 or 3 tons. Crushing is done in three stages and reduces the ore to about 10 per cent plus  $\frac{1}{2}$ -in. and before the treatment process it is screened on 20-mesh screens. About 20 per cent of the total *caliche* to the plant is removed as fines for treatment in a filter plant and 80 per cent of the ore remains as coarser material for vat leaching.

In the filter plant—of the Moore leaf type—the fines are mixed, 2 tons of fines to 1 cu. m. of hot mother liquor to make a pulp of 60° C. and 1.8 density. The pulp is then ready for filtering, and when cakes 1 to  $1\frac{1}{2}$  in. thick are formed the filter baskets, each having a capacity of 25 tons of cake, are transferred to a brine-wash tank for a displacement wash. Water cannot be used because of serious cake pitting and therefore poor washing. The final washed filter cakes contain 3 to 4 per cent nitrate (mostly due to the nitrate content of the brine used for wash) and are discharged into water and pumped to waste.

The coarse *caliche* is delivered into large concrete vats (from 7000 to 12,000 metric tons capacity) for leaching. There are ten of these vats, to provide time for loading, unloading, the four-stage leaching cycle, washing and draining.

The leaching cycle consists of downward percolation of solutions, adding mother liquor to the vat in the fourth leaching stage and advancing the solution through vats in third, second and first-stage leaching to produce a strong solution. The total time of contact of the *caliche* with the leach solution is about 80 hr. In contrast to the Shanks process, the

leaching temperature is around 40° C. and the nitrate pickup from mother liquor to strong solution is from 330 to 450 grams per liter. Space here does not permit a discussion of the influence of other salts in the *caliche* on the leaching and crystallizing process, except to say that temperature has but little influence on the solubility of sodium chloride and sodium sulphate, so that when a strong saturated nitrate solution is cooled sodium nitrate is precipitated, leaving practically all other salts in solution.

After leaching with "mother liquor," the residue is washed with a series of graduated washes and finally with water. Extra water is always added to produce the brine wash for use in the filter plant. The vat residue containing 1.0 to 1.5 per cent nitrate is then unloaded mechanically, using 5-ton clamshell grab buckets, into 16-ton dump cars and is hauled to waste.

The strong solution from the vat leaching and the filter-plant leaching is combined and pumped to the crystallizing plant, which consists of a series of 20 shell and tube tanks, each containing six hundred 3-in. vertical tubes 16 ft. long, and the nitrate solutions flow from one tank to another with gradual cooling from 40° to 10° C. The solution to be cooled is caused to circulate rapidly through the inside of the vertical tubes. The cooling medium is on the outside of the tubes. The first 14 tanks of the series of 20 tanks are used as recuperators—as the cooling medium is the 10° C. mother liquor which re-enters tank No. 14 and emerges from tank No. 1 at 35° C.—countercurrent cooling. The last six tanks are used as refrigerators and the cooling medium is liquid ammonia. Five 300-ton (ice-rating) York ammonia compressors service these six refrigerators.

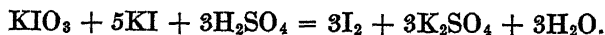
Crystallized nitrate sludge from the crystallizers is delivered to a battery of 24 centrifuges for dewatering. The product is pure white, about 48 mesh in crystal size, and is of 95 to 96 per cent purity with 3.0 to 3.5 per cent moisture.

While "mother liquor" from the crystallizing plant emerges at 35° C., leaching temperatures throughout the plant are at 40° C. The extra heat required to heat the mother liquor, as well as the *caliche*, and to provide for heat losses from the large open vats, is obtained as waste heat from the Diesel power plant.

Of the total heat content of the fuel oil used in the 21,000-hp. Maria Elena and 28,000-hp. Pedro de Valdivia power plants, about 33 per cent is converted to electrical energy, and a large part of the remaining heat that goes into the Diesel cooling-water system and exhaust gases is delivered to the nitrate solutions through a system of heat interchangers. In all, about 80 per cent of the total heat content of the fuel oil is recovered either as electrical energy or as heat for the leach solutions. Even the ammonia condensers are cooled with leach solutions to recover the heat removed in the crystallizing plant.

In order to present the Guggenheim process nitrate to the customer in a superior form, the centrifuge nitrate is granulated. The granulation process consists in melting the centrifuge nitrate in suitable oil-fired furnaces and spraying the melt at 340° C. into a large chamber. The liquid droplets, in their passage through the air, cool below the freezing point and are collected as pearl-like pellets of  $\frac{1}{32}$  to  $\frac{1}{8}$ -in. diameter on a belt conveyer underneath the spray chamber, and then are conveyed to coolers and finally to a storage bin for bagging or bulk shipment, as required. The nitrate so produced has a purity of about 98.5 per cent and contains small amounts of sodium chloride, sodium sulphate, borax, potassium, perchlorate and iodine (as iodate) and other salts essential for the development of plant life.

*Iodine.*—Iodine as an iodate dissolves in the nitrate leach solutions and builds up to concentrations of from 5 to 10 grams per liter. By the addition of sodium bisulphite (a local product made from sulphur dioxide and soda ash) the iodate is reduced to iodide. Then exactly  $\frac{1}{5}$  more of the original solution containing iodate is added, and solid iodine is formed, which settles to the bottom of the reaction tanks:



The spent liquor is returned to the nitrate plant and the iodine sludge is drawn off, filtered, pressed into "cheese cake" and retorted, delivering a crude product consisting of blue-black, lustrous crystals of about 99.4 per cent purity. This product is usually sublimed by purchasers for delivery to consumers. The iodine for export is packed in small wooden kegs of 70-kg. capacity and is covered with rawhide before shipment.

*Sodium Sulphate.*—Sodium sulphate is produced by leaching the *caliche* residue in the vats with water after the nitrate has been extracted. This solution will contain at 20° C. about 125 grams per liter sodium sulphate, and when refrigerated to 0° C. deposits sodium sulphate decahydrate, and leaves a solution with 45 grams per liter sodium sulphate. The decahydrate is centrifuged and delivered to a dehydrating plant, which consists of an oil-fired spray tower, a crystal settler, a filter and a rotary drier. A stream of sulphate solution circulates through the spray tower and crystal settler, at about 70° C. The decahydrate is delivered into this stream of hot solution, and as the melting point of the decahydrate is 33° C., the salt is promptly converted to a strong sulphate solution, which is subsequently evaporated in the spray tower. The volume of circulating solution is constant and evaporation made up by the decahydrate additions. The anhydrous sulphate sludge is partly dewatered in the filter, and finally dried in the rotary drier.

The product is 97 per cent in purity with small amounts of moisture—sodium chloride and sodium nitrate. The chief market for this product is in the Kraft paper industry.

## TESTS AND SPECIFICATIONS, MARKETING AND USES

Chilean nitrate, in accordance with the specifications laid down by the Sales Corporation, must contain a minimum of 95.5 per cent sodium nitrate and a maximum of 2 per cent moisture, 1.5 per cent sodium chloride, 0.75 per cent potassium chloride and 0.5 per cent borax. Chilean nitrate shipped to markets contains from 15.5 to 16.2 per cent nitrogen, and in addition contains the principal vital rare elements magnesium, iodine, boron, calcium, potassium, sodium, lithium and strontium, which are so desirable for the proper development of plant life.

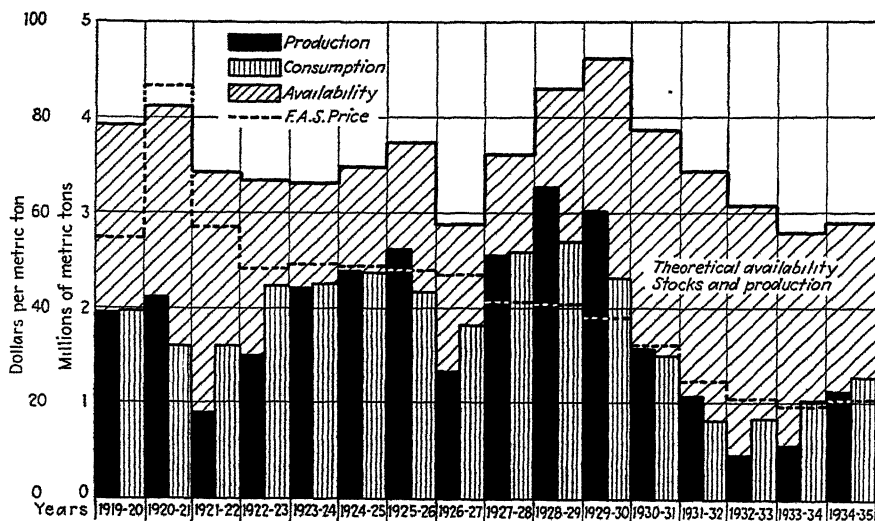


FIG. 5.—AVAILABILITY, PRODUCTION, CONSUMPTION AND PRICE OF CHILEAN NITRATE.

Chilean nitrate is sold all over the world, the principal consumers being the United States, Egypt, Spain, Germany, France, Holland, Belgium, Sweden, Denmark, Japan, Italy, Czechoslovakia, the Baltic States, Peru and the Argentine. Shipments are made from Chile by steamer to the principal ports of consuming countries and distributed to dealers and consumers. The principal uses of sodium nitrate are as fertilizer and in the manufacture of chemicals and explosives.

Variation in prices is shown in Fig. 5.

## ACKNOWLEDGMENT

The author wishes to thank Mr. P. F. Kruger, General Administrator in North Chile; Mr. G. F. Coope, formerly Assistant Administrator, and Dr. E. S. Freed, Research Chemist of the Lautaro Nitrate Co., Ltd. and the Compañía Salitrera Anglo-Chilena, for aid in compilation of the data in this chapter.

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## CHAPTER XXXIII

### PHOSPHATE ROCK

BY H. S. MARTIN\* AND JAMES WILDING,† MEMBERS A.I.M.E.

THE enormous importance of phosphates to agriculture is shown by the fact that during the 10 years ending with December 1936 the world's production of high-grade calcium phosphate minerals was nearly 94,100,000 metric tons,<sup>9</sup> much the greater part of which was used as fertilizer. Only a small proportion was used for metallurgical purposes and the manufacture of elementary phosphorus and chemicals.

Most of this large tonnage was consumed in Europe and the United States, an increasing proportion being taken by Japan, Australia, New Zealand, Egypt and other countries.

During the last 75-year period the mining of phosphates has been carried on for varying lengths of time in most of the countries of Europe but, owing to exhaustion of ore and to increasing competition of American and North African mineral, has gradually ceased except in Belgium, Esthonia, France, Poland, Russia and Spain. The present European production, except in Russia, is now of minor importance. Phosphates are at present mined on a major scale in the United States, Morocco, Algeria, Tunisia, Egypt, Russia and a few islands in the Pacific.

In the eighteenth century a phosphatic greensand from deposits in Cambridgeshire was used in the east of England as a fertilizer. It was not until after the researches of Justus von Liebig in the third decade of the nineteenth century that it was realized that the beneficial effect of the greensand was due to its contained phosphate of lime. Liebig showed (ref. 5, p. 591) that phosphates, potassium compounds and other mineral elements must be supplied to the soil, in order that its fertility should be kept up, and made up artificial manures of these compounds with ammonium salts. His investigations on the subject were first published in 1840 in his book, *Die Chemie in ihrer Anwendung auf Agricultur und Physiologie*. Liebig was closely followed by John Lawes, who began his agricultural experiments at Rothamsted in 1837. In 1842 Lawes took out a patent (ref. 5, p. 301) for an artificial manure made by treating

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The geologists of the United States Geological Survey have written a series of very instructive monographs on the phosphate deposits of the United States. The information given in those publications has been freely used in this chapter, especially, with regard to the deposits of the Rocky Mountains region.

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mineral phosphates with sulphuric acid, thus initiating the superphosphate industry, since grown to such great importance. In conjunction with J. H. Gilbert, Lawes continued his agricultural investigations at his experimental farm at Rothamsted until he died in 1900.

#### COMPOSITION, PROPERTIES AND ORIGIN

The phosphate minerals of economic importance contain calcium tribasic phosphate as principal constituent. They may be classed as apatite, a macrocrystalline mineral of very widespread occurrence in igneous and metamorphic rocks, and the cryptocrystalline, amorphous or submicrocrystalline minerals which occur in sedimentary rocks of various geological periods.

Apatite crystallizes in the hexagonal system, often in long prismatic forms. The composition of the mineral of commercial apatite deposits is  $\text{Ca}_{10}\text{F}_2(\text{PO}_4)_6$  (fluorapatite). In some deposits a part of the fluorine is replaced by chlorine but in none is chlorapatite the important mineral. The specific gravity of apatite is approximately 3.2, its hardness 4.5 to 5.0, its color usually greenish, sometimes white, gray or brown. It is translucent to opaque and of a subresinous to a stony luster. The few minable deposits known are in metamorphic rocks<sup>30</sup> and are, perhaps more generally, held to be due to magmatic differentiation, though there are differing opinions as to their origin. An exception to the above statement is the occurrence in the Caceres district of Spain of massive or fibrous apatite in limestone near to granite.

The calcium phosphate mineral of the sedimentary deposits, from which comes the great part of the phosphates of commerce, is usually amorphous so far as the eye can discern even with microscopical aid (collophane of Austin), but it is sometimes cryptocrystalline. Recent research work by several investigators<sup>3,10,11</sup> has shown that the minerals are really crystalline and give the X-ray diffraction pattern of fluorapatite, hydroxyapatite, staffelite and dahlite. In staffelite and dahlite the fluorine of fluorapatite is partly replaced by the carbonate radical; in hydroxyapatite all or part of the fluorine may be replaced by OH. This explains the variability of the composition of the phosphate mineral, which may be considerable even in the same deposit. In North American deposits the ratio of fluorine to phosphorus pentoxide is higher than in apatite but no free fluorite can be identified. In some of the deposits of the Pacific Islands, notably in those of the West Indian Islands, the ratio is lower than in apatite. The investigators of the United States Department of Agriculture<sup>12</sup> have stated that the phosphate of all commercial varieties of phosphate rock mined throughout the world is an apatite-type mineral and mostly is fluorapatite or hydroxyfluorapatite.

The hardness and specific gravity of the phosphate mineral of the sedimentary deposits vary somewhat but both are less than the corre-

sponding properties of macrocrystalline apatite. The color of the phosphate varies from white, through gray and brown to almost black, the dark color often connotating a higher content of organic matter than is found in the mineral of lighter color.

The sedimentary deposits are of marine origin. Some, as laid down, were rich in phosphate or have become so by subsequent subaqueous action and have not been further enriched since emergence from the sea. Some were originally phosphatic limestones or marls from which calcium carbonate was dissolved away by waters containing carbon dioxide, in which calcium carbonate is much more soluble than calcium phosphate, leaving a residue correspondingly enriched in the latter. This enrichment may have been increased by the interchange of dissolved phosphate ions in the waters for carbonate ions of the rock mineral. Some deposits may be due to the transportation by flowing water of phosphatic residues from the weathering of surface rocks and their re-deposition with other debris in shallow coastal waters. Yet other deposits may have been formed on beaches as the result of the disintegration of phosphate-bearing cliffs and the sorting action of waves. More than one of these agencies may have contributed to the formation of any deposit, now sufficiently high in calcium phosphate to be an economical source thereof.

The work of the investigators of the Challenger and later ocean research expeditions has shown the presence of nodules or concretions of calcium phosphate minerals in the muds and oozes of the ocean bottom,<sup>6,22</sup> especially on the continental shelf. They have also shown the presence of phosphoric acid in sea water from traces to three parts of phosphorus pentoxide per million of water (ref. 22, p. 185). The phosphoric acid (or dissolved phosphate salts) is considered to be chiefly derived from decaying remains of marine fauna, which in certain parts are found in great accumulations on the sea bottom. At or near such accumulations phosphate nodules are especially abundant.

## DISTRIBUTION OF DEPOSITS

### UNITED STATES

There are no large deposits of apatite in the United States. A very small tonnage is mined at Piney River, Virginia, where it occurs with ilmenite and rutile. It is used for making phosphate salts.

Deposits of sedimentary origin, which have yielded phosphate rock in commercial quantity, occur in 12 states of the Union. Deposits which have not so far been mined occur in 8 other states. In 6 of the 12 states mining has only been intermittent and the total tonnage from each occurrence unimportant. The states in which the important deposits occur are South Carolina, Florida, Tennessee, Idaho, Montana, Wyoming and Utah.

*South Carolina.*—The first phosphate mining in the United States was done in South Carolina in 1867. The deposits are confined to the seaboard zone, extending from Beaufort to somewhat northeast of Charleston, a distance of about 70 miles. There are two types of deposits, land rock and river rock.

The land rock occurs<sup>27</sup> in a bed varying in thickness up to 30 in. It is a phosphatic residue of a marl of the Miocene period. It consists of an indurated layer of phosphate mineral, honeycombed with cavities filled with calcareous sandy clay. The mineral is associated with a dark sandy clay in which numerous phosphate nodules are embedded. In most places, where mined, the bed was less than 18 ft. below the surface.

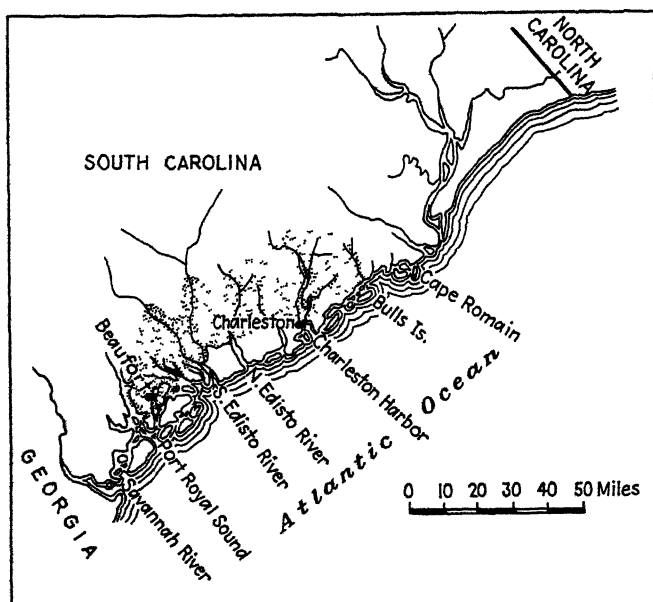


FIG. 1.—COAST REGION OF SOUTH CAROLINA. PHOSPHATE DEPOSITS OCCUR IN SHADED AREA.

The river rock occurs in loose rounded fragments that have accumulated in irregular banks on the bottoms of streams which flow through the land-rock areas. In places it occurs in thin beds like the land rock.

The mining of the land rock in early years was by hand labor; in later years of exploitation the overburden was removed by steam shovel and the rock mined by hand labor. Later still, both overburden and rock were removed by steam shovel. In early days the river rock was recovered by negro divers and later by dredges.

The rock was crushed and cleaned by washing in log washers or in rolling cylinders and on screens by high-pressure water sprays.

The analyses of the shipments varied considerably (Table 1).

The peak of South Carolina production was 541,645 tons in 1889. It gradually fell off and ceased altogether in 1920, owing to the increasing competition of the more cheaply mined and higher grade Florida phosphate. The production of river rock ceased in 1909.

The total recorded production from 1867 to 1920 was more than 13,000,000 tons of cleaned mineral. It is estimated that there are reserves of 9,000,000 tons.

*Florida.*—Deposits of phosphates occur mostly on the west of the longitudinal axis of the peninsula from near the northern end to somewhat more than halfway down. The largest deposits, beds of so-called "land-pebble" phosphate, occur in Polk and Hillsborough Counties,

TABLE 1.—*Analyses of Rock*

	Land Rock	River Rock
Calcium tribasic phosphate, per cent. ....	58 -63	53 -62
Calcium carbonate, equivalent to the contained carbon dioxide, per cent. ....	7 -14	3.8-11
Iron oxide and alumina, per cent. ....	1.5- 4	
Insoluble, per cent. ....	8 -13	9 -24
Organic matter, per cent. ....	3.5- 6.5	

where they are extensively mined. "Hard-rock" deposits occur in the more northern counties, the more important being in Marion, Citrus and Hernando Counties, 70 to 100 miles north of the land-pebble district.

The land-pebble deposits occur in an area about 30 miles long by 30 wide.<sup>13</sup> They are unconsolidated or very slightly consolidated sediments that are considered to have been formed by the disintegrating and sorting effect of ocean waves acting on phosphatic coastal rocks of an earlier period. In them are fossil teeth and bones of both land and marine animals—sharks' teeth are common—indicating that the sediments have lain at some period under very shallow water (coastal swamps) and at another period have been submerged under a considerable depth before being covered with their overburden of sand and clay.

The phosphate beds lie almost horizontally, show distinct stratification and consist of a matrix of sand, clay and soft white crumbly phosphate mineral, in which are embedded rounded or subangular fragments of harder phosphate, ranging in size from about 2 in. to fine sand. The pebbles form from 10 to 50 per cent of the bed. The beds are from 3 to 18 ft. thick and are overlain by loose or slightly cemented sand and clay, varying in thickness from a few feet to 40 ft. or more.

The hard-rock deposits (ref. 13, p. 5) are considered to be replacements of the calcium carbonate of limestone beds by phosphate dissolved out of phosphatic residues of overlying beds and, in places, to be reprecipitations of such dissolved phosphate in open cavities in phosphatic lime-

stone. The thickness of the phosphate beds varies from a few feet to a maximum of 100 ft. The phosphate mineral occurs in the form of rock fragments, boulders, plate rock, in a matrix of sand, clay and soft white phosphate, the hard rock forming up to 25 or 30 per cent of the bed. There occur also in the beds boulders of flint and fragments of limestone sometimes partly changed to phosphate. The thicknesses of the overburden ranges from a few feet to 50 ft. It consists of coarse, angular, loose or slightly indurated sand, separated from the matrix by a thin layer of bluish clay.

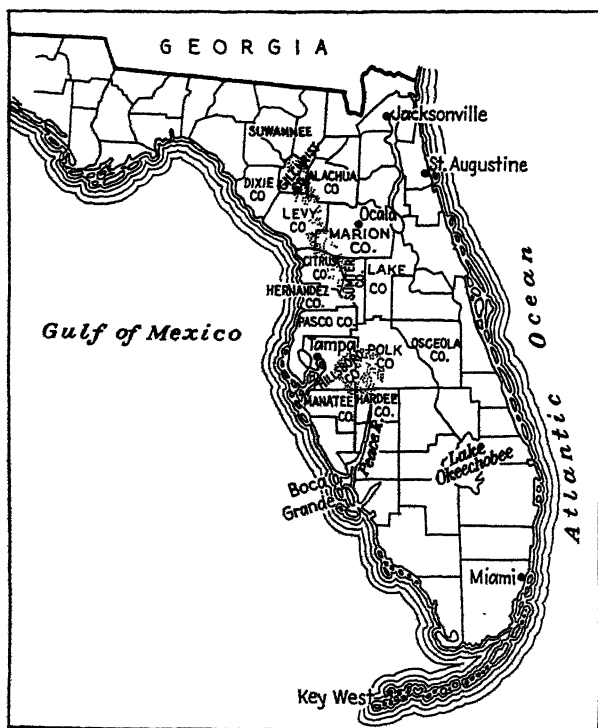


FIG. 2.—PHOSPHATE-PRODUCING AREAS (SHADED) IN FLORIDA.

Phosphate mining began in Florida in the year 1888 with the dredging of concentrations of the mineral from the bed of the Peace River. River mining ceased in 1908 or 1909. From 1888 to the end of 1936 the total production of cleaned phosphate mineral has amounted to about 79,500,000 tons. The greatest production was 4,500,000 tons in 1930.

Analyses of the cleaned mineral shipped from the land-pebble area vary from 66 to 77 per cent  $\text{Ca}_3(\text{PO}_4)_2$ , 1.5 to 3 per cent  $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ , 6 to 10 per cent  $\text{SiO}_2$  and 0.3 per cent organic matter. The hard-rock shipments are higher in grade, 78 to 83 per cent  $\text{Ca}_3(\text{PO}_4)_2$ .

A few years ago reserves were estimated at 97,000,000 tons of high-grade phosphate mineral. Since the introduction of flotation treatment of the fines, which formerly went to waste, they are estimated at some 236,000,000 tons on a basis of 70 per cent grade. Areas not formerly considered profitable have become available.

*Arkansas.*—There are estimated to be upwards of 10,000,000 tons of 40 per cent rock in the State of Arkansas.

*Tennessee.*—Important deposits of phosphates occur in a district about 60 miles S.W. to S. by W. of Nashville, in the south central part of the state. Those at present being mined in Giles and Maury Counties belong to either of two types of deposits, distinguished as brown rock and blue rock.

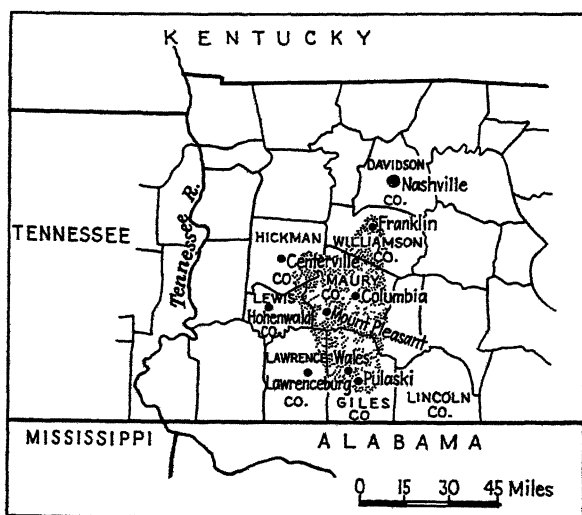


FIG. 3.—PHOSPHATE DEPOSITS BEING WORKED IN CENTRAL TENNESSEE (SHADED).

Brown-rock deposits<sup>29</sup> are considered to be the residual product of the weathering and leaching of the outcrops of phosphatic limestone beds or the beds themselves where exposed on the dip. The phosphatization of the outcrops does not penetrate very far into the strata in the rim deposits, not more than 60 ft., and the zone of change to the unaffected limestone is usually abrupt or very short.

The phosphate mineral occurs in coherent plates and loose grains associated usually with much clay and a little sand. (Plate rock about 15 per cent of the total phosphate; remainder small pebble and sandy mineral.) The deposits are from 2 to 10 ft. thick (a few are much thicker in places) and are nearly horizontal or have the gentle dip of the underlying limestone. They outcrop on steep hillsides or are exposed on the dip or are covered with an overburden of chert, clay and soil, which seldom exceeds 20 ft. in thickness.

The blue phosphate rock is an unaltered, highly phosphatic bed of Mississippian age. In thickness it varies from a few inches to 3 or 4 ft.

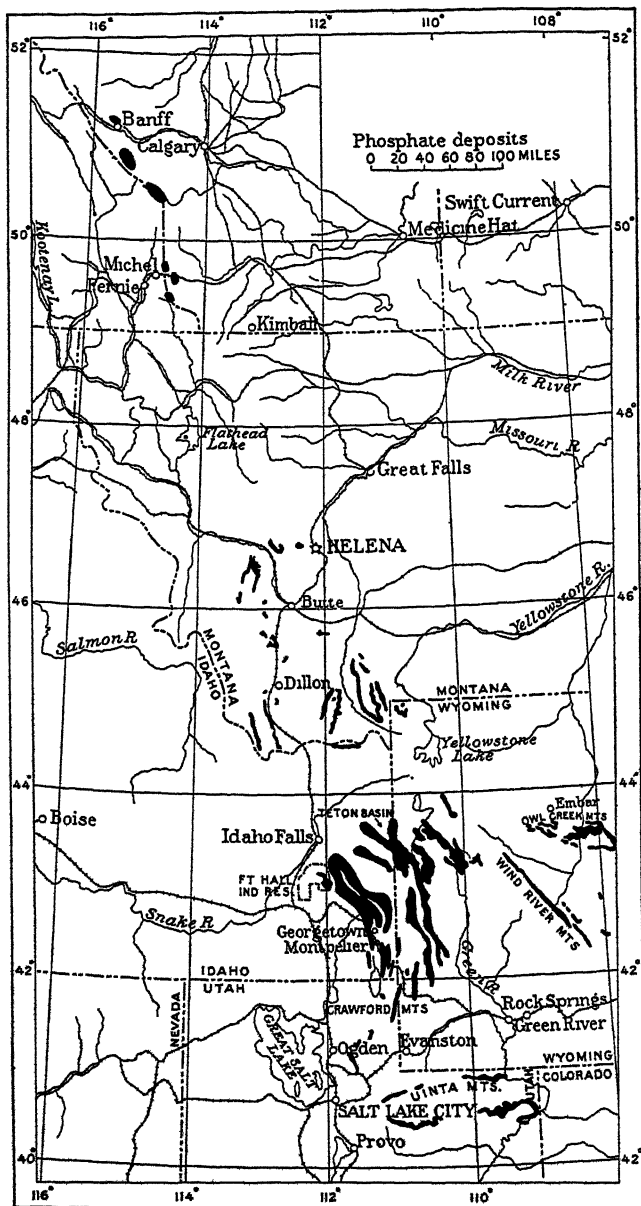


FIG. 4.—LOCATION OF KNOWN PHOSPHATE-BEARING AREAS OF PERMIAN AGE IN THE ROCKY MOUNTAINS. (FROM MANSFIELD.<sup>18</sup>)

and in structure is shaly, conglomeratic or compact. Where compact, it may contain up to 75 per cent  $\text{Ca}_3(\text{PO}_4)_2$ . Where shaly or conglomeratic,

it is much lower in grade. In the compact variety phosphate grains are sometimes cemented together by chalcedony and pyrite. The beds outcrop in the walls of stream valleys.

Phosphate mining began in Tennessee in 1894 and to the end of 1936 the total production of cleaned mineral was about 18,879,000 tons. The greatest production of any year was 633,939 tons in 1929. The reserves are estimated at some 18,000,000 tons.

Analyses of the cleaned mineral vary from 70 to 78 per cent  $\text{Ca}_3(\text{PO}_4)_2$ , 3 to 6 per cent  $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ , 3 to 5 per cent  $\text{SiO}_2$  and 0.35 per cent organic matter.

### *Rocky Mountain Region*

In southeastern Idaho, southwestern Montana, western Wyoming and northeastern Utah, there is a series of sandstones, limestones and shales considered to be of Permian age. The United States geologists have named them Phosphoria Series because of the occurrence in them of strata high in phosphate mineral. The series is from 100 to 300 ft. thick and the phosphate strata occur at two principal horizons that are 20 to 60 ft. apart. The region has suffered prolonged erosion and the Phosphoria Series outcrops at many places on the flanks of mountain ranges. The phosphate beds are of marine origin and it is the opinion of geologists that they have not undergone much concentration by terrestrial agencies.

The phosphate rock has much the same character over the whole region. It is oolitic in structure, the mineral being cryptocrystalline. In color it varies from gray to brown or black. A characteristic is the fetid odor of a freshly broken surface. The ores of the region as mined are higher in calcium phosphate, lower in iron, alumina and carbonate and much lower in silica than those of Florida.

The minable beds are from 4 to 10 ft. thick and dip at angles up to  $55^\circ$ . Some mining has been done by stripping of overburden and open-cut methods but most is by underground work. A considerable proportion of the available rock lies above the natural drainage horizon.

*Idaho.*—The important deposits occur in the counties of Bear Lake, Bingham, Bonneville, Caribou, Teton and Madison, in the southeast corner of the state. So far, production has come mainly from mines in Bear Lake and Caribou Counties, not far from the Wyoming border. Seven companies have properties in these two counties,<sup>15</sup> all of which are within 8 miles of the Oregon Short Line. The most important mine is at Conda, Caribou County. It belongs to the Anaconda Copper Mining Co., which has equipped it for mining on a large scale, made roads and built numerous houses for its employees. The ore is shipped to the phosphate plant at Anaconda.

Mining began in 1907 near Montpelier in Bear Lake County but has not been continuous. Since the start the total phosphate mineral



production of the state up to the end of 1936 has been 709,000 tons.<sup>17</sup> The production of 1936 was 56,300 tons. The reserves are estimated at an enormous figure.

*Montana.*—Phosphate deposits occur somewhat west of the Continental Divide,<sup>23</sup> near Elliston and Garrison in Powell County, Philipsburg in Granite County, Melrose in Silver Bow County,<sup>26</sup> Three Forks in Gallatin County<sup>4</sup> and Dillon in Beaverhead County. The fields are all near to or tapped by railroads.

Mining began in 1921 and has been carried on in the Philipsburg, Garrison and Melrose fields, but in some years little or nothing has been done. Most of the Garrison ore is shipped to the phosphate plant of the Consolidated Mining and Smelting Co. at Trail, B. C.

The total production of the state up to the end of 1936 has been 151,200 tons. The production in 1936 was 28,200 tons. Estimates of reserves of 97,000,000 tons have been made for the Garrison field; of 86,000,000 tons for the Elliston field. The reserves of the other fields are very extensive but estimate figures have not been made for them, as insufficient work has been done.

*Utah.*—Phosphate deposits occur on the north and south slopes of the Uinta Mountains in the counties of Summit, Daggett, Wasatch, Duchesne and Uinta and also in Weber, Morgan and Rich Counties, not far from the Wyoming line.

Some mining was done as early as 1907 or 1908 but so far there has been little development. The occurrences are not within easy reach of the railroad. There has been no production of recent years and up to the end of 1935 the total production of the state had reached only some 20,000 tons. The reserves, however, are large, estimates of 96,000,000 tons having been made for two areas only. The few figures for grade that we have found seem to show that the deposits are of considerably lower phosphate content than those of Idaho and Montana.

*Wyoming.*—Phosphate deposits occur on the slopes of the Wind River Mountains and the Owl Creek Mountains in Fremont County and also in the Salt River Range and the Beckwith Hills in Lincoln County, not far from the Idaho line.

Although some mining had been done before the war, the total production of the state up to the end of 1935 has been unimportant, only 61,270 tons. There has been no production during the last few years. The reserves of three or four areas have been estimated at 37,000,000 tons. Not sufficient work has been done to justify figures for the other known occurrences.

*Shipments and Producers.*—In the early days of mining in the Rocky Mountain region a great part of the production was shipped through Pacific ports to Australia and Japan but these exportations ceased after the development of the deposits on islands in the Pacific Ocean. Of

recent years most of the Idaho and Montana ore has gone to the phosphate plants of the Anaconda Copper Mining Co. at Anaconda and the Consolidated Mining and Smelting Co. of Canada at Trail, B. C., to be converted into phosphoric acid and double and triple "super."

The companies that own properties and are mining or have mined in the region are:<sup>21</sup> Anaconda Copper Mining Co., Conda, Caribou County, Idaho; Solar Development Co. (subsidiary of Consolidated Mining & Smelting Co. of Canada), Bear Lake County, Idaho; Agricultural Potassium-Phosphate Co. of California; San Francisco Chemical Co., Montpelier, Bear Lake County, Idaho; Bear Lake Phosphate Co., Paris, Bear Lake County, Idaho; Idaho Phosphate Co., Paris, Bear Lake County, Idaho; Utah Fertilizer & Chemical Co., Georgetown, Bear Lake County, Idaho; Montana Phosphate Co., Philipsburg, Granite County, Mont.; Dissett Bros., Garrison, Powell County, Montana.

Mining in the region has so far been on privately owned land but much of the phosphate area in the four states is Federal land.<sup>15,21,23</sup> The United States Government has withdrawn from entry 970,460 acres in Idaho, 119,230 acres in Montana, 302,470 acres in Utah and 1,019,170 acres in Wyoming. The Government grants leases of areas up to a maximum of 2560 acres on the following terms:

First year.....	25 cents an acre
Second to fifth year, inclusive .....	50 cents an acre
Sixth year and onward.....	\$1.00 an acre

A royalty of 2 per cent of the gross value of the material produced is also charged.

### *Reserves*

The deposits of the four Rocky Mountain States retain their thickness and quality over large areas and are considered to be original sediments and not of secondary formation. Upon these facts and upon the correlation of strata by geologists at many outcrops rest the enormous estimates of reserves in the region. Of high-grade material alone (over 58 per cent  $\text{Ca}_3(\text{PO}_4)_2$ ) nearly six billion tons is thought to be available, of which some five billion are in Idaho. The reserves will certainly last many generations.

From what has been said it would appear that within the boundaries of the United States there exists enough phosphate mineral to supply the whole world for six or seven hundred years at the present rate of consumption. Not all of it will be as easily mined as the deposits of Florida and Tennessee. The deposits of the Rocky Mountain States will probably not be extensively exploited until mining becomes more costly in Florida and Tennessee through the exhaustion of the more easily mined and better grade areas.

## CANADA

In eastern Canada there are deposits of apatite (which were reported in 1848 by T. Sterry Hunt) north of Ottawa in the region of the Lievre and Gastineau Rivers in Quebec and west of Ottawa in several counties of eastern Ontario. The apatite occurs in irregular pockets,<sup>30</sup> sometimes connected together, in a series of pyroxenic rocks. It is associated with mica, pyroxene, sometimes with minor amounts of sphene, scapolite, zircon, pyrite and pyrrhotite. There is usually no sharp separation of wall rock and mineral, the bodies appearing to be due to magmatic differentiation. However, there is considerable difference of opinion as to the origin of the deposits. The apatite is usually coarsely crystalline and of a greenish color.

Some mining was done in the sixties of the last century and there was an irregular small production from 1870 to 1894, the peak year being 1890, when somewhat less than 32,000 tons was shipped. The ore as shipped ran from 80 to 88 per cent  $\text{Ca}_3(\text{PO}_4)_2$ .<sup>\*</sup> Mining of phosphate ceased because the ore cost too much to compete with that from South Carolina and Florida. Prices rose irregularly from \$10 in 1871 to \$21 per ton in 1883, falling quickly thereafter to \$6 in 1894. It is believed that deposits are by no means worked out but no estimate of reserves can be made.

The first ore mined was turned into superphosphate at Brockville, Ont. Later some ore was shipped to the United States and Germany but most of it went to Great Britain.

In western Canada, in the mountain region between Banff and Crow's Nest Pass, there are sedimentary phosphate deposits that are, perhaps, of the same geological age as those of the Rocky Mountain region of the United States and of similar origin. Some ore has been mined in the Crow's Nest district by the Consolidated Mining & Smelting Co. of Canada, but the deposits do not seem to be of commercial importance.

## EUROPE

*Norway.*—Apatite deposits are found in metamorphic rocks in the coastal zone from 100 to 150 miles south-southwest of Oslo.

In the veins the apatite is associated with mica, pyroxene, feldspar, tourmaline, rutile, titanite and pyrite. Deposits were mined at Kragerö as early as 1854 and later at Oedegården, but the output was always small and mining ceased in 1926. Up to that year the total Norwegian production had been about 153,000 tons. The material shipped was of high grade—up to 90 per cent calcium phosphate (the content of pure fluorapatite is 92.2 per cent). The reserves are of very limited extent.

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<sup>\*</sup> The small production of recent years is the by-product of mica mining.

*Russia.*—Near to Khibinogorsk, on the Kola Peninsula, on the west side of the White Sea, there are exceedingly large deposits of apatite.<sup>14</sup> The best deposits occur in the Kukisvum mountain at an altitude of 250 to 450 meters above the sea. The deposits are in nephelite-syenite rock and the ore itself is mainly a mixture of apatite and nephelite, the accessory minerals, aegirite (= acmite), magnetite and titanomagnetite only amounting to from 5 to 20 per cent of the whole.

The upper part of the ore, 50 or 60 m. thick, is stated to contain 67 per cent of calcium phosphate on the average; the lower part, 130 to 140 m. thick, 49 per cent on the average.

Mining began in 1930, ore containing 50 to 55 per cent calcium tribasic phosphate being shipped. Since the middle of 1931 an increasing proportion of the ore has been concentrated to more than 80 per cent calcium phosphate but considerable crude ore is still shipped, most of which goes to Germany.

The upper high-grade portion of the deposits is estimated to contain 60 million tons of calcium tribasic phosphate; the lower and lower grade portion, 60 million tons. Other deposits of the region, not yet exploited, are estimated to contain 500 million tons of the phosphate.

In a number of places in European Russia there are sedimentary phosphate deposits, many of them being in the region between the Volga and Dnieper Rivers. Large deposits occur in western Siberia, in the provinces of Uralsk and Turgai.

Dr. Khvostovsky, writing in 1931,<sup>14</sup> states that by the end of 1933, the production of phosphates during the first Five-Year plan would be:

	TONS
Central Asia (Aktubinsk* phosphates).....	8,500,000
Central U.S.S.R.....	5,500,000
Urals.....	475,000
Khibinsk apatite.....	1,500,000
Total.....	15,975,000

\* Aktubinsk or Aktiubinsk is said by the geographical authority of the New York Public Library to be in southwest Siberia. This is confirmed by a map of Siberia, which shows the place at the extreme west of Turgai. As far as we have been able to find out the production during the five-year period was somewhat more than 2,000,000 tons, and the total production up to the end of 1935 about 5,000,000 tons. The output for 1936 was 1,497,800 tons.

Professor Pranishnikov estimates the phosphate mineral reserves of Russia at 5,563,000,000 tons. We presume that represents ore in the ground, the grade of which may be very imperfectly known. The geologists of the U.S.S.R. believe that their country contains 62 per cent of the phosphate resources of the world.

*Germany.*—Sedimentary phosphate deposits occur in Nassau in limestone. A number of years ago there was a more or less regular production from mines in the region. Shipments ran from 60 to 80 per

cent calcium phosphate and sometimes were high in carbonate. In some years 30,000 tons was mined. There has been no mining for many years but the present German Government is said to be studying the possibility of restarting mining in the hope of cutting down the present large imports of phosphates.

*Poland.*—During the last eight years there has been produced in Poland about 106,000 tons of phosphate rock. The deposits lie near Grodno, about 95 miles northeast of Warsaw, and near Niezvisk, about 300 miles southwest of that town. The greatest production was 39,300 tons in 1929. The production in 1936 was 15,500 tons. Reserves now are estimated at 369,000,000 tons.

*Belgium.*—Phosphate deposits have been mined in Belgium since the early seventies of the last century. They occur in the Mons and Liège districts. The deposits occur in beds and pockets in the chalk and calcium phosphate mineral is associated with much carbonate in the Mons district and with much siliceous material in the Liège district.<sup>24</sup> The rock is enriched by sorting and washing operations and in some mines by pneumatic separation, but, as shipped, is not of high grade, containing around 60 per cent of calcium phosphate.

The Belgian production before the war was about 200,000 tons a year. In 1935 it was 26,500 tons. The total production up to the end of 1936 has been about 9,150,000 tons. The reserves are limited and of low grade.

*France.*—Deposits of phosphate rock occur in numerous places in northeastern France, in south central France and in the Pyrenees region. In the northeastern district the phosphate rock occurs in pockets of limited extent and in beds in phosphatic chalk. The phosphate of the pockets is mostly in the form of unconsolidated sandy material; the phosphate of the beds occurs in nodules of calcium phosphate in a stiff clay. The beds contain 35 to 40 per cent of nodules. The material shipped contains from 50 to 75 per cent calcium phosphate, 5 to 12 per cent calcium carbonate, 1 to 20 per cent silica.<sup>24</sup>

Where the ore occurs in beds, the surprising feature is the thinness of the beds that have been worked—6 to 8 in. The ore is sorted by hand and some of it is enriched by washing or pneumatic concentration.

In the south central district (Quercy) phosphate rock occurs in large pockets in limestone. Those more or less circular vary in diameter up to 35 m.; the elongated ones have dimensions of from 3 to 6 m. wide by up to 90 m. long. The pockets pinch out at comparatively shallow depths.

The material mined runs usually over 70 per cent calcium phosphate, about 7 to 8 per cent carbonate, and is low in silica, but contains 4 to 5 per cent iron and alumina.

Mining of phosphates has been carried on in France since the late fifties of the last century. The production rose to 536,000 tons in 1901

but decreased thereafter. Since the war the production has gone steadily down, that of 1936 having been only 33,000 tons. The total production since the beginning of mining operations has been more than thirteen and a half million tons. Reserves of good grade are of limited extent.

*Spain.*—Phosphate deposits in the province of Caceres occurs in several places.<sup>28</sup> Apatite occurs in well defined bodies in granite at Malpartida de Caceres and was mined for a number of years. The mineral is crystalline, and, as shipped, contained from 85 to 90 per cent of calcium phosphate. In the best years only 6000 tons was shipped.

At Logrosan phosphate mineral occurs in veins in slate and quartzite. The mineral is subcrystalline and is associated with quartz. The ore, as shipped, contained from 80 to 85 per cent of calcium phosphate.

Near to Caceres phosphate occurs in pockets and veins in limestone and slate. It is massive and amorphous, sometimes dense and sometimes granular and friable. It is high in calcium carbonate.

Phosphates have been mined in Spain since 1860. In early years the ore was shipped to England.

The Spanish production has never been great, the total since mining began up to the end of 1935 having been about 800,000 tons. The production in 1936 was 8000 tons. The production of any year has not much exceeded 40,000 tons.

In a paper read before the International Geological Congress, in 1926, C. Rubio and J. de Gorostizaga<sup>28</sup> give the Spanish reserves of 60 per cent calcium phosphate and upwards as: In the Province of Caceres—proven 1,800,000 tons; probable 14,000,000 tons and, also, large tonnages of lower grade ore; in the province of Murcia, 40,000,000 tons of the lower grade ore. Of the last not much is known.

#### NORTH AFRICA

*Morocco.*<sup>1</sup>—Attention was drawn to the existence of phosphates in Morocco in 1908 by A. Brives, in a communication to the Académie des Sciences. Exploration of the deposits was delayed until 1917. The deposits occur in a zone lying at a distance of 100 to 200 km. from the stretch of coast between the ports of Rabat and Safi, at the western end of the northern flank of the Atlas mountains. Six areas in the zone have been studied and mining has been carried on in two of them—Ouled Abdoun since 1921 and Ganntour since 1929 or 1930.

The deposits occur in a series of limestones, marls and clays (of the Eocene period) which have been subjected to much erosion, so that outcrops of phosphate beds have been traced on the flanks of the hills for long distances. There are three and in places four beds of phosphate of from 1 to 2½ m. thick. The phosphate content of the beds is from 48 per cent to more than 76 per cent calcium phosphate. Only the deposits of over 70 per cent have been exploited up to the present but

methods of concentration of the lower grade material are being investigated. The diluent is mostly calcium carbonate.

The phosphate beds of the Ouled-Abdoun and Ganntour districts lie not far from horizontally. Mining is done through adits and drifts by a system of rooms and pillars, the workings being allowed to cave after removal of the ore. Not much timbering is required and much of the timber used is recovered.

The ore, as mined, resembles a wet beach sand. It contains about

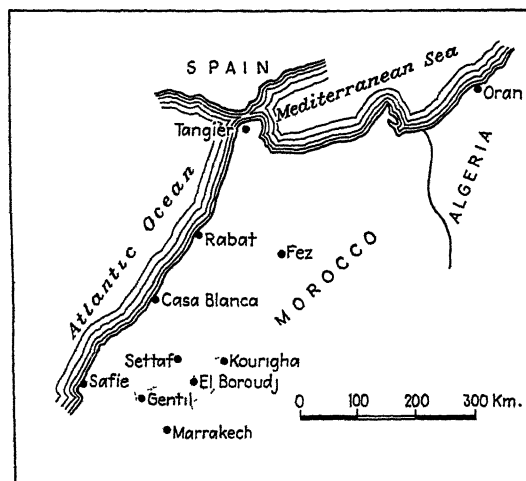


FIG. 5.—PHOSPHATE REGIONS OF MOROCCO (SHADED).

10 per cent water. The waste (flint, limestone, marl) is much greater in size than the phosphate and is separated on sieves of 2-cm. aperture. A second screening on sieves of 8 mm. aperture is done after drying. The drying and screening is the only preparation given the ore.

The mining center for the Ouled-Abdoun area is Kourigha; that for the Ganntour area, Louis Gentil. In each of these centers a church, school, post office, administration building, drying plants and dwellings have been built.

The mined ore is transported to Kourigha or to Louis Gentil to be dried, either in the sun in the dry season, or in drying furnaces. For drying in the sun the ore is spread out in shallow layers and turned over mechanically. The moisture is decreased from 10 to 2.5 per cent. The drying and screening equipment at each place has a capacity of 2000 tons in 10 hr. The Kourigha ore is shipped through the port of Casa Blanca; the Gentil ore through the port of Safi.

The total production from the beginning of mining to the end of 1936 has been about 15,047,800 tons. It was highest in 1930, being 1,779,000 tons; in 1936 it was 1,257,800 tons.

The deposits are considered original sediments and the beds of even tenor. The reserves are thought to be enormous but the Office Cherifien des Phosphates has only concerned itself with the deposits of greater than 75 per cent calcium phosphate. For these it gives figures of 110 to 130 million tons.

*Algeria-Tunisia.*—The phosphate deposits of economic interest in Algeria and Tunisia are sedimentary beds in the lower Eocene. They occur in two important groups, in zones near to the Algeria-Tunisia border but mostly on the Tunisian side. In the west of the Department

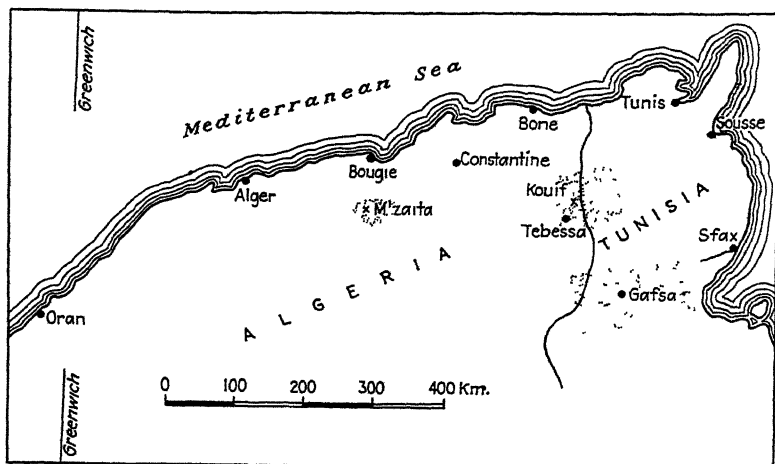


FIG. 6.—PHOSPHATE REGIONS OF ALGERIA-TUNISIA.

of Constantine in Algeria are other less important deposits. The existence of phosphate deposits in Algeria and Tunisia has been known since 1873, but there was no exploitation until 1893.

The deposits are beds interstratified with a series of limestones and marls, with some conglomerates. In each mining locality several phosphatic beds are known but only one to three of them contain enough phosphate to be worth mining under present conditions. It is not profitable to mine ore of less than 59 per cent calcium phosphate, owing to the low price it commands in the market.

The thickness of the beds that are mined is from 1 to 4½ m. In the west of Constantine beds of less than one meter are mined. On Djebel Onk, a range of hills of the Gafsa region, but on the Algerian side of the border, there are veins as much as 35 m. thick with rock of from 58 to 60 per cent calcium phosphate. These deposits have been studied but no mining operations have been undertaken as yet. It is hoped to mine a great part of the ore by open cut.

The dip of the beds in the different localities varies from low angles to 70° and even 90°. Generally the country is deeply cut by gorges, ravines and valleys and the phosphate beds outcrop over long stretches.



Some ore is extracted by open-cut work but mostly it is mined through adits, the beds being cut by inclined winzes into blocks 10 to 15 m. wide. The hanging wall is supported by timbering as stoping advances from one level to the next above; when the ore is extracted, the roof is allowed to cave.

The phosphate mineral of the Algerian and Tunisian deposits is oolitic in structure, the oolites, of 0.05 to 0.5 mm. diameter, contain about 70 per cent calcium phosphate. They are cemented together by a phosphatic matrix, which runs about 25 per cent calcium phosphate. In the southern area the cement is softer than the oolites; in the northern area it is much harder than in the south. In three of the mines of the southern area advantage is taken of the relative softness of the cement to enrich the mined rock. The rock is carefully crushed and the resulting more finely crushed poor matrix is rejected by screening and pneumatic separation. The concentration effected is at best some four to six units. In other cases the ore is dried in the sun or in furnaces and shipped without further treatment.

The most important deposits are those of the Gafsa region, in the South of Tunisia, which occur over an area about 175 km. from east to west and about 75 km. from north to south. Its central point is the oasis of Gafsa, about 175 km. west southwest of Sfax, the port on the Tunisian coast through which most of the rock is shipped. A small portion of this phosphate area is on the Algerian side of the border. The phosphate beds are in anticlinal hills, the axes of which are east and west. The region has not suffered much from erosion and the available reserves are believed to be milliards of tons.<sup>1</sup>

The rock shipped from the Gafsa region contains from 58 to 66 per cent calcium phosphate, 8 to 15 per cent calcium carbonate, 5 to 6 per cent calcium sulphate and is low in silica, ferric oxide, alumina and fluorine.

The deposits of the Tebessa region are some 100 km. north of the Gafsa region, on both sides of the Algeria-Tunisian border, within an area of approximately 80 km. east to west by 70 km. north to south.

The phosphate beds are in synclines, the upper portion of which has been eroded away. The region has suffered much erosion.

The largest mine is that of Kouif on the Algerian side of the border. It is equipped to produce 700,000 tons annually. Liquid oxygen is used for blasting in the mine. The ore is crushed, sorted and dried before shipment. It runs from 65 to 72 per cent calcium phosphate, 12 to 25 per cent calcium carbonate, 3 to 4 per cent calcium sulphate and is low in ferric oxide, alumina and silica.

The reserves of rock of sufficiently good grade to be minable under present conditions are limited, perhaps 20,000,000 tons.

The deposits of the west of Constantine in Algeria are in the high

plateau region southwest of the port of Bougie. The plateau has been much cut up by erosion, and there are numerous small faults in the deposits. Mining began in the region in 1902 and has been carried on in four localities but continues at present in only one. The ores shipped run from 58 to 65 per cent calcium phosphate, 10 to 15 per cent calcium carbonate, 3 to 4 per cent calcium sulphate, 10 per cent silica and are low in ferric oxide and alumina.

The important companies that have produced or still are producing in Tunisia and Algeria are: Compagnie des Phosphates et du Chemin de Fer de Gafsa; Compagnie Tunisienne de Phosphates du Djebel M'Dilla; Société des Phosphates Tunisiens, Compagnie des Phosphates de Constantine, Compagnie de Saint Gobain.

Where mining has ceased, it has only in a few instances been due to exhaustion of the deposits; usually to the difficulty of marketing at a profit the comparatively low-grade ore. The main diluents of the product are carbonate and to a lesser degree sulphate, which are not easily separated from the phosphate mineral either by gravity or flotation methods. There is also the difficulty of lack of water. It has been proposed to calcine the rock, which is high in carbonate, at a temperature high enough to drive off the carbon dioxide ( $600^{\circ}$  to  $700^{\circ}$  C.) and to slake and flush away with water the resulting lime from the undecomposed phosphate. This can be done easily, but whether it would be a profitable method of concentration for any ore except those very high in carbonate, is, perhaps, doubtful.

The total reserves in the Algerian-Tunisian fields<sup>1</sup> are very large, some 1,350,000,000 tons, of which about one-fourth is in Algeria and three-fourths in Tunisia. The average grade of the reserves is about 61 per cent calcium phosphate. These are the reserves of the known deposits. There are "probable" reserves of 10,000,000,000 tons in Tunisia. The production in 1936 was 2,281,200 tons.

*Egypt.*—Phosphates occur in the Nile region and near the Red Sea. Mining in the Nile region, never important, has ceased, but two companies are operating near Kosseir and Safaga, through which ports the rock is shipped. The phosphate mineral occurs in thin beds interstratified with marls, limestones and cherts of the cretaceous formation. The ore runs from 63 to 70 per cent calcium phosphate, is high in carbonate and low in silica. The mines are within 26 miles of the coast and are connected with the ports by narrow-gauge railways. (Fig. 7.)

The operating company at Kosseir is Italian, the Società Egiziana per Fosfati et Extrazioni; that at Safaga is British, the Egyptian Phosphate Co. Since the beginning of mining, the production in Egypt has been about 4,600,000 tons. The peak year was 1936, when the production was 498,560 tons. More than half the production of the Red Sea companies goes to Japan.

*West Indies.*—Phosphates have been mined in several of the West Indian Islands, the chief producers having been Sombrero, Aruba and Curaçoa. It has ceased because of exhaustion of deposits except in Curaçoa. The Curaçoa production during the 10 years ending with 1936 has been 899,300 tons. The grade is high, 75 to 85 per cent calcium phosphate, with low ferric oxide and alumina. The reserves are limited.

*Asia.*—Phosphate deposits have been mined in China, Netherlands Indies, Indo-China, British India and Japan but, except in Japan, the production has been quite unimportant. Production began in Japan in 1905 but was inconsiderable until after the war. During the 10 years ending with 1935 the total production has been 400,000 or 450,000 tons.

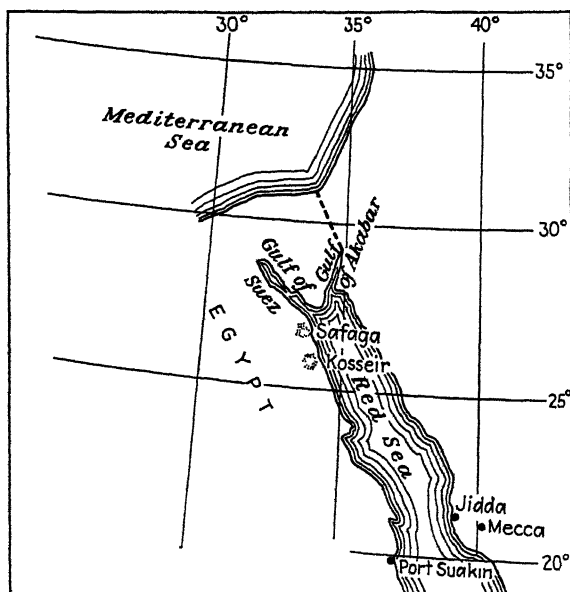


FIG. 7.—PHOSPHATE DEPOSITS OF RED SEA AREA (SHADED).

*Islands of the Pacific.*—Phosphate deposits are known in many of the islands of the Pacific Ocean and are mined in quantity on a few. The most important deposits are on Nauru Island, midway between the Marshall and Solomon groups, practically on the equator; on Ocean Island, 250 miles east by south of Nauru, but considered a western outlier of the Gilbert group; Angaur, in the West Carolines group, formerly Pellew group; Makatea in the Paumoto or Tuamoto archipelago, 135 miles north of Tahiti.

Nauru is an island of about 5000 acres in area, the central part of which rises to about 250 ft. above sea level. This part is a mass of phosphate rock of considerable depth. The island was formerly German territory but is now under the joint mandate of Great Britain, Australia

and New Zealand. Mining began in 1907 and was carried on before the war by a company in which the Pacific Phosphate Co. (British) held a considerable interest. The ore is of very high grade, shipments running 85 to 89 per cent calcium phosphate. Most of the ore is shipped to Japan, Australia and New Zealand.

On Ocean Island occurs a deposit similar to that of Nauru Island. Before the war it was worked by the Pacific Phosphate Co. After the war the British, Australian and New Zealand Governments bought the mining rights of the company in both Nauru and Ocean Islands. Mining is now under the supervision of a commission appointed by the three governments.

Mining began on Ocean Island in 1902, on Nauru in 1907. The reserves are estimated at from 100 to 120 million tons for the two islands together. The total production for the 10 years ending with 1936 has been 6,013,100 tons. The production in 1936 was 476,100 tons.

Angaur Island (with Peleliu or Peririju) was formerly German territory but now is under the mandate of Japan. Mining began on Angaur in 1909 and rose to 90,000 tons before the war. The ore is high grade, the shipments containing about 80 per cent calcium phosphate. The total production of Angaur for the period 1927 to 1936, inclusive, was 670,400 tons. It was all shipped to Japan. The Japanese also mine deposits on the Japanese islands, Daitajima and Rasa, about 500 miles south of Japan. The total production of Peleliu, Daitajima and Rasa in 1936 was 110,680 tons.

The reserves of the four islands<sup>21</sup> are estimated at 2,600,000 tons, of which nearly three-fourths is on Angaur. The grade of the reserves is between 70 and 85 per cent of calcium phosphate.

Makatea Island is French territory. It has an area of some 3000 to 3500 acres and rises to a level of 230 ft. above the sea. The phosphate deposits are worked by the Compagnie Française des Phosphates. Mining began in 1911 or 1912 and during the period 1927 to 1936, inclusive, there was mined 1,288,600 tons; the greatest output of any year, that of 1936, was 130,770 tons. The ore is of high grade, 80 to 85 per cent calcium phosphate, the ferric oxide and alumina are low, the silica very low. Much the greater part of the output goes to Japan.

The reserves are estimated at 25 to 30 million tons.

An important producer of phosphates is Christmas Island, in the eastern part of the Indian Ocean, about 190 miles south of Java. The island is about 12 miles long and 9 miles wide and rises to a height of 1000 ft. above the sea. Phosphates are mined in the rounded hills of the higher part of the island.

Mining began in 1899; the production reached 290,000 tons in 1907 and during the period 1927 to 1936 inclusive was 1,315,517 tons, a great part of which has gone to Australia and Japan. The rock is of high

grade, containing from 80 to 86 per cent of calcium phosphate, 6 to 7 per cent of calcium carbonate. It is very low in ferric oxide and alumina and very low in silica.

### POLITICAL CONTROL

The control of the phosphate rock output of the world is in the hands of private corporations except in the following instances:

The deposits of Soviet Russia are entirely controlled by the State.

The deposits of the islands under the mandate of Japan are controlled by the South Seas Mandate Bureau of the Japanese Department of Overseas Affairs.

The deposits of Ocean and Nauru Island are controlled by a commission appointed by the Governments of Great Britain, Australia and New Zealand.

The Moroccan deposits are owned by the Moroccan State, and are controlled by L'Office Cherifien des Phosphates, a commission appointed by the State. Morocco is a protectorate of France and the commission must guide itself by the conditions of the Treaty of Algeciras, 1905, which prescribe that no nation may be favored economically over others in Moroccan Commerce.

The deposits of Algeria and Tunisia are controlled by private companies but we understand that, owing to the rapid expansion of production in Morocco in the latter part of the last decade, the French Government took some action by way of limiting output in both Algeria-Tunisia and Morocco to prevent too intense competition.

Since the Moroccan production became so great, the proportion of the total European consumption supplied by Florida has considerably decreased though, owing to increase of consumption, the actual Florida tonnage shipped to Europe is not much less than formerly.

### PRODUCTION AND CONSUMPTION

The production of high-grade phosphate material in the United States<sup>25</sup> during the 10 years ending with the year 1936 was 30,542,000 tons, an average of 3,054,200 tons per year. The greatest production of any year was 3,989,200 in 1930; the smallest production was that of 1932—1,738,400 tons. The production of 1936 was 3,323,800 tons. The world production of the same 10-year period\* was 94,096,000 tons, an average of 9,409,600 tons per year. The greatest production of any year was 11,656,400 tons in 1930; the smallest 7,068,598 tons in 1932. The production of 1936 was 10,494,286 tons.

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\* There is some doubt about the world figures, as the Russian figures included therein may be of Khibinsk apatite only. If so, the world production figures should be increased considerably.

The proportion of the world production of 1935 consumed by various countries is given below:

	Per Cent		Per Cent
United States .. . . .	20.47	South Africa .. . . .	1.05
Japan. . . . .	10.29	North Africa.. . . .	0.98
Russia . . . . .	8.53	Poland .. . . .	0.68
France .. . . .	8.44	Latvia and Estonia ..	0.65
Germany.....	8.17	Canada.. . . .	0.55
Spain.....	7.03	Hungary . . . . .	0.45
Italy....	7.01	Rumania and Greece . .	0.41
Australia.....	4.81	Finland .. . . .	0.32
United Kingdom.. . .	4.76	Austria .. . . .	0.16
Holland....	3.77	Norway .. . . .	0.16
Belgium. . . . .	2.38	Switzerland.. . . .	0.13
New Zealand . . . .	2.28	Danzig .. . . .	0.11
Denmark. . . . .	2.08	Yugoslavia .. . . .	0.10
Sweden... . . . .	1.60	Hawaii.. . . .	0.06
Portugal ... . . . .	1.22	Various. ....	0.14
Czechoslovakia.....	1.21		

The correctness of the consumption figures is somewhat vitiated by the fact that several of the countries export manufactured phosphate products.

The United States is much the largest consumer of phosphates but if the consumption is expressed in quantity of fertilizer phosphoric acid (phosphorus pentoxide) per unit of cultivated area it does not rank high. In this respect Japan holds the first place, followed by Holland, Belgium, Denmark, Germany, Great Britain, Spain, Australia and New Zealand. Not far from half of Germany's consumption of phosphoric acid is in basic (Thomas) slag, which, of course, is not derived from phosphate rock. Other European countries also use considerable basic slag; the United States but little.

#### PROSPECTING, EXPLORATION AND MINING

In the Florida and Tennessee fields prospecting is usually done by drilling holes with a 4-in. auger. For rough exploration these holes are drilled 4, 8 or 16 to the 40-acre tract. A log is kept of the formations that the auger passes through, the prospector determining at what depth the auger passes through the overburden and into the phosphate matrix. By observation the experienced prospector determines when the auger has passed through the bed of matrix and into the barren bedrock under the matrix. The depth of the matrix determines the volume that each prospect hole represents. Sometimes the matrix is estimated to weigh 90 lb. per cu. ft. dry, and sometimes the deposit is opened up at several

points and the actual weight of a cubic foot in place is determined. Such prospect samples of the matrix are washed free of clay in the laboratory and an estimate made of the amount of available rock in the deslimed material. At the present time, amount of washer rock is obtained by screening on a sieve of 14 to 28 meshes per inch, and the finer material ranging from 14 to 150-mesh size treated by laboratory flotation tests in order to determine the approximate yield of flotation concentrate from such areas.<sup>3</sup>

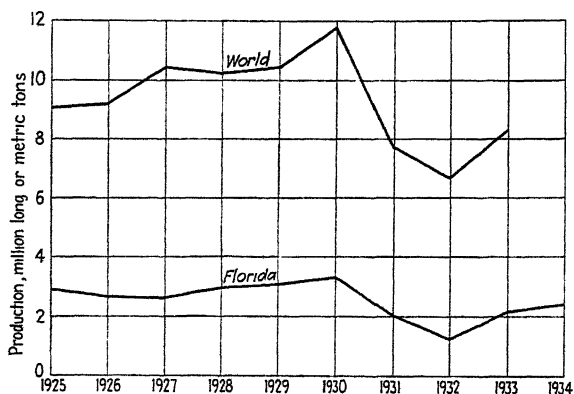


FIG. 8.—PRODUCTION OF PHOSPHATE ROCK.

Until the flotation process was developed the tonnage of rock per acre was calculated from the percentage weight of material that would remain on the sieve used, and no account was made of the fine rock that passed through the sieve, because this material was then wasted by the washers. In the pebble district, the finer material represents, on the average, as large a tonnage of rock as the coarse fractions, so that the present method of prospecting doubles the estimated tonnage per acre. Many deposits that were unworkable a few years ago on account of the low yield of washer-size rock can now be mined economically, using flotation concentration on the finer portion.

The western phosphate deposits are prospected by the usual underground mining methods, consisting of tunnels, or shafts with drifts, and crosscuts.

*Mining Methods.*—The Florida pebble phosphate consists of more or less horizontal layers of phosphate matrix overlain by varying depths of overburden. The overburden is removed usually by electric dragline, although sometimes hydraulically, and the mining of the matrix is done by hydraulic means. As the mining is carried on at present, the overburden ranges from 8 to 30 ft. in depth and the matrix from 4 to 18 ft. The dragline strips off the overburden from a cut 150 to 225 ft. wide, the overburden being dumped back into the adjacent mined-out cut, except

when the first opening is made. The matrix is then broken up by two or three hydraulic guns, using nozzles  $1\frac{1}{2}$  to 2 in. in diameter and water under a pressure of 200 lb. per sq. in. Ditches and a "well" are cut into bottom rock and the slurry of matrix is picked up by sand pumps and pumped to the washer. The pumps are 10, 12, or 14-in. size. In average matrix this method moves 200 to 300 cu. yd. of matrix per hour. In deep, sandy matrix, as much as 400 yd. per hour may be mined. About 4000 gal. of water per minute is used by the hydraulic guns.

In the Tennessee field and in one or two locations in Florida, the mining and stripping are both done by dragline or steam shovel and the matrix conveyed to the washers by dump cars. In the hard-rock field of Florida most of the mining is done by steam-shovel dipper mounted on a floating steel scow, the wet matrix being conveyed to the washer by a cable-operated skip.

At Pembroke, Polk County, Florida, the Phosphate Recovery Corporation is mining some old washer-waste dumps, using a suction dredge. On a wooden scow 40 by 20 ft. is mounted a 10-in. Georgia Iron Works sand pump with a flexible suction that can be raised and lowered by winch. A cutter head and high-pressure nozzle are used to cut through strata of clay present in the dump. This method is successful and cheap on the washer-waste dumps, but when tried on original matrix years ago proved a failure.

#### PREPARATION FOR MARKET, TESTS AND SPECIFICATIONS

In the Florida pebble field the slurry of phosphate matrix pumped to the washer is partly dewatered on slotted screens and the dewatered material is delivered to a series of log washers and screens. The clay is removed from the phosphate particles by the disintegrating action of the log washers and high-pressure sprays, and a size separation is made of the phosphate pebbles from the material, depending on the point at which quartz begins to degrade the product. The size of screen opening to be used in the washer is determined by a screen analysis of the deslimed matrix. As a rule, quartz particles begin to degrade the phosphate at the minus 14 plus 20-mesh fraction, but in certain areas the grade does not drop until the minus 20 plus 35-mesh fraction is reached. Most operators adjust washer screens to give about 6 per cent acid-insoluble matter in the finished rock. Until 1927 that part of the matrix of particle size less than 14 or sometimes 20-mesh, was waste and not utilized. Since 1927, flotation concentration of this portion of the matrix has developed rapidly and there are seven plants in Florida concentrating this fraction by flotation or tabling.

The minus 14-mesh washer waste is pumped to settling bins, where the finest part of the material (usually the minus 150-mesh portion) is overflowed to waste. Sometimes rod mills are used to grind the larger



particles of the washer waste to approximately minus 28-mesh, and after such grinding and desliming, the material is conditioned by agitation with a fatty acid, fuel oil and caustic soda. The conditioned pulp is then either passed over vibrating tables or into froth-flotation machines, where a separation is effected between the phosphate and quartz particles. A flowsheet of the typical flotation plant for treating the washer waste is

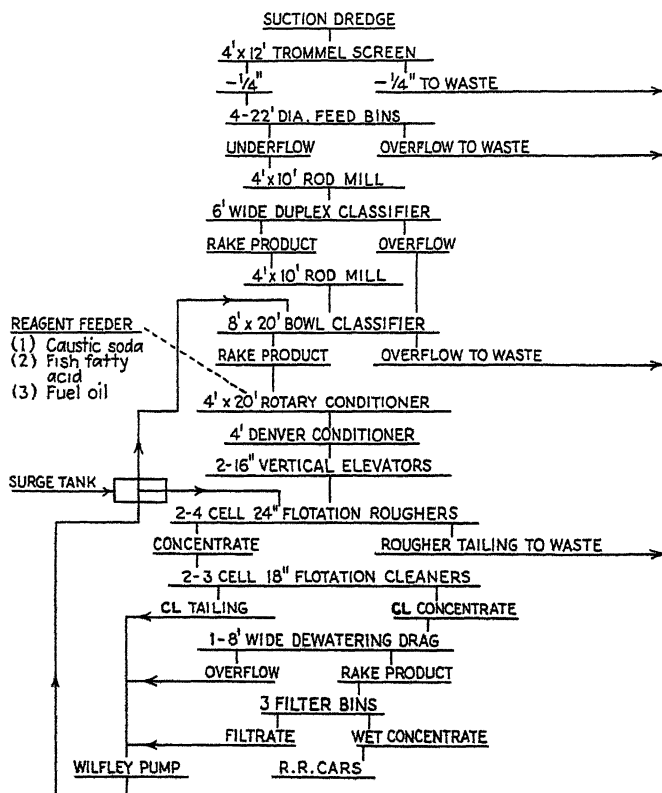


FIG. 9.—FLOWSHEET OF PLANT 4, PHOSPHATE RECOVERY CORPORATION, PEMBROKE, FLORIDA.

shown in Fig. 9.<sup>19</sup> Both washer rock and flotation concentrate are hauled either by trolley or railroad cars to a central drying plant, where the material is dried in rotary oil-fired kilns before final shipment.<sup>2</sup>

As to the simple method of flotation treatment described in the foregoing, it should be said that the flotation man knows that for metallurgical results it is not the best possible. Optimum recovery and grade are not to be effected by treating in one operation all sizes of material from the limiting coarse size to fines. For best recovery and at the same time best grade of concentrate, either of two methods can be used: After a first flotation, the coarser material may be separated from the

tailing and re-treated or the flotation plant feed itself may be divided into two or three sizes to be treated separately. As the phosphate mineral and the gangue differ so little in specific gravity, the necessary sizing can best be done by classifying methods. That such procedure is not adopted generally is due to the effort to keep down costs, even at some sacrifice of metallurgical results. The phosphate operator is handicapped by the extremely low price of his product.

Phosphate rock is sold principally on the tricalcium phosphate (or B.P.L.) content of the rock. There is also a guarantee made as to maximum amount of iron and alumina present. The lower grades are sold on a guarantee of 4 per cent maximum iron plus alumina and the higher grades on a 3 per cent iron and alumina guarantee. The commercial grades sold by the Florida pebble-phosphate producers range from 68 to 77 per cent bone phosphate of lime. Where the rock is to be used in reducing furnaces for the production of phosphorus and phosphorus pentoxide coarser material is demanded, and various consumers have certain size specifications, although in recent years the finer flotation concentrate is in great demand by fertilizer manufacturers because of small particle size and ease of fine grinding. Some rock is calcined before shipping, for special purposes. Calcination removes organic matter and makes it easier to produce water-white phosphoric acid by the wet method.

#### MARKETING, USES AND PRICE

By far the larger part of the phosphate rock produced is used in making fertilizer, principally superphosphate, but smaller quantities are used in the production of elemental phosphorus, phosphoric acid, trisodium phosphate, dicalcium phosphate, and other phosphorous chemicals. Some colloidal phosphate and some finely ground rock is used for direct application to the soil, and a small amount is used in stock food.

Exports of phosphate from the United States are handled through an organization known as the Phosphate Export Association and the domestic marketing is done by individual contracts principally. In the Florida pebble phosphate field there are at present seven operators producing pebble phosphate rock. Of these producers, four companies manufacture and sell commercial mixed fertilizers, using part of their rock in their own fertilizer plants.

Average sale prices of Florida phosphate have ranged from a maximum of \$5.38 per long ton in 1921 down to \$2.91 in 1933. Florida hard-rock phosphate has ranged from \$11.31 per long ton in 1920 to \$5.75 per long ton in 1934. During the latter part of 1935 the domestic prices were cut to the lowest figure in the history of phosphate rock. At the present time prices for various grades range from \$1.75 to \$3.50 per long ton.

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## CHAPTER XXXIV

### POTASH

BY HOWARD I. SMITH,\* MEMBER A.I.M.E.

**P**OTASH, which is of prime importance in agriculture as a plant food and is widely used in the chemical industry, derives its name from the iron pots used to evaporate solutions leached from plant ashes. The product was mostly an impure potassium carbonate. Later the word was used to include caustic potash obtained from treating pot ashes with lime. It is still used as a general term in agriculture and industry for various compounds of potassium, but where so used in this chapter and expressed as percentage of potash or pure potash, it applies to the theoretical equivalent in potassium oxide ( $K_2O$ )—a compound never found in nature or manufactured in commerce but generally used as a common denominator for comparison of all potassium compounds. The making of potash from plant ashes is a very old industry, but potassium in mineral form was first noted in 1797, when it was identified in leucite.<sup>3</sup> The first person to recognize the importance to plants of potash in the soil was von Liebig,<sup>3</sup> whose published reference thereto appeared in 1840, three years before the discovery of potash in the brine of a well drilled for salt near Stassfurt.

#### HISTORY OF AMERICAN POTASH INDUSTRY

Among the colonists reaching Virginia in 1608 were men from Poland skilled in making potash from wood ashes.<sup>7</sup> At different times after 1635, when the American product entered the trade, the production of potash in this country was subsidized or tariffs were imposed. This industry reached its peak in 1825 and rapidly declined after the Civil War, when the forests in the areas most productive of potash became depleted, and labor became more profitably employed, with the improvement of transportation facilities that permitted the marketing of timber, farm products and livestock. A contributing factor in the decline was the substitution, beginning in 1823, of cheaper sodium carbonate made by the Le Blanc process, for much of the potash used in industry. Another

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<sup>3</sup> References are at the end of the chapter.

contributing factor was the discovery of natural potash salts in Germany, and their development in 1861. An educational program devised to stimulate the use of potash as a fertilizer soon followed this development, and the production from natural deposits increased rapidly. With cheap soda ash and German potash available, the interest of the American people in a domestic source of potash dwindled. However, interest was reawakened in 1910, when certain contracts for the purchase of foreign potash that were favorable to American fertilizer manufacturers were canceled, and the extent of American dependence on foreign potash, particularly for agriculture, became realized. Therefore an extensive search was begun in 1911 by several Government agencies,<sup>20</sup> which, together with those of private interests, led directly to the establishment of the present industry in America, chiefly at Searles Lake, Calif., and in Eddy County, New Mexico.

#### COMPOSITION AND PROPERTIES

The principal potash mineral mined throughout the world is sylvite, or muriate of potash ( $\text{KCl}$ ). Pure sylvite contains the equivalent of 63.2 per cent  $\text{K}_2\text{O}$ , the highest percentage of all the potassium minerals.

Carnallite ( $\text{KCl} \cdot \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ), when pure, contains the equivalent of 16.9 per cent  $\text{K}_2\text{O}$ , the lowest in pure potash of the salts mined in quantity. It was the first potash salt produced from saline deposits and still ranks second in tonnage. It is seldom worked except in combination with sylvite or for valuable byproducts such as bromine and magnesium chloride.

Kainite ( $\text{MgSO}_4 \cdot \text{KCl} \cdot 3\text{H}_2\text{O}$ ), containing 18.9 per cent  $\text{K}_2\text{O}$ , was the second of the soluble potash minerals worked in Germany. It is still mined in small amounts in Europe but has little relation to the kainite of commerce, a low-grade potash fertilizer containing the equivalent of 14 to 20 per cent  $\text{K}_2\text{O}$ .

Langbeinite ( $2\text{MgSO}_4 \cdot \text{K}_2\text{SO}_4$ ), containing the equivalent of 22.7 per cent  $\text{K}_2\text{O}$ , formerly mined in Germany is not now produced to any extent except in Poland. Deposits containing a high percentage of langbeinite have been located by core drilling in New Mexico.

Polyhalite ( $\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 2\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), containing 15.6 per cent  $\text{K}_2\text{O}$ , found at Ischl, Austria, and described<sup>8</sup> in 1820, was the first potash mineral associated with the salt deposits in New Mexico to be identified. It has been mined to a small extent in Germany, but if any is produced today it is an inconsequential quantity that may occur with other potash minerals.

Halite ( $\text{NaCl}$ ) or common salt is the principal impurity occurring with potash salts in saline deposits.

Kieserite ( $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ ) often occurs with potash salts. Some is sold in crude form for fertilizers and other quantities are used for other purposes.

Many other potash minerals have been found in the Permian salts but they are of secondary importance commercially. Leonite ( $K_2SO_4 \cdot MgSO_4 \cdot 4H_2O$ ) and aphthitalite (glaserite)  $[(K,Na)_2SO_4]$  have been identified from American sources. Glaserite is one of the principal potash minerals in the Searles Lake deposit.

Natural brines in the Dead Sea in Palestine and in the spongelike mass of salt in Searles Lake, Calif., are important sources of potash.

Orthoclase ( $K_2O \cdot Al_2O_3 \cdot 6SiO_2$ ), containing 16.9 per cent pure potash when not replaced by sodium, is the principal source of potash in the ceramic industry.

Glauconite, leucite and alunite, formerly considered important natural sources of potash in America, are abundant, but of little prospective value compared to the New Mexico deposits, unless they can be made to yield other products at a profit, along with the potash.

Sylvite is the only one of the soluble potash minerals now being mined in the United States except as others occur with it in quantities too small to be seen in the salt as mined. Its color ranges from clear to milky white and brick red. Many of the clear or milky white crystals are surrounded by reddish brown sylvite, which, when dissolved in water, leaves as a residue a reddish brown to milky flocculent substance resembling algae in form. This substance may also occur along the cleavage planes of clear crystals. Small quantities of blue halite are usually found in association with sylvite and rarely elsewhere. The origin of the coloring has not been determined.

Sylvite has a hardness of 2.2 and can be scratched with the thumb more easily than halite. It is very plastic under pressure, and with unrestricted sides a halite-sylvite mixture, such as is now being mined in New Mexico, can be compressed 60 per cent and still maintain the approximate initial bearing strength per square inch. This is an important factor in mining and must be considered in selecting fine-grinding equipment. The sylvite ore contains so much water that a considerable amount is squeezed out if the ore is compressed. When the ore is dissolved, bubbles of gas, probably nitrogen, are given off.

Crystals of sylvite are isometric. Its specific gravity is 1.98, luster vitreous, and cleavage cubical. The taste, often reported as bitter, is in reality salty and similar to that of halite. Sylvite is more soluble in warm water than halite and hence has a quicker or sharper taste. It loses more heat than halite in dissolving and therefore has a greater cooling effect on the tongue. After tasting several pieces, however, it is difficult to distinguish sylvite from halite by this means.

Langbeinite is isometric. It ranges from clear to gray, its hardness is 4.2, specific gravity 2.82, luster vitreous and fracture conchoidal. It has no cleavage. After exposure to the atmosphere for some time, as in a core box, the outside of the specimen is altered to a white powder, prob-

ably leonite and epsomite, bringing out the triangular shape of the crystals in contrast to those of the associated halite or anhydrite. It is tasteless, somewhat granular and very slowly soluble in water.

Polyhalite is triclinic. It ranges in color from white to gray, salmon, and dark brick red. It is normally opaque and rarely translucent. Its hardness is 3.6, specific gravity 2.77, and luster dull. It is massive and breaks with a conchoidal fracture. Coarsely ground material is very slowly decomposed by water at ordinary temperature. Polyhalite has no taste. It can be distinguished from anhydrite by its appearance after wetting and drying. The water dissolves part of the potassium and magnesium sulphates and a coating of white gypsum is left on the polyhalite specimen. When anhydrate is similarly wetted it is not dissolved and on drying retains its natural color.

Carnallite is orthorhombic. It ranges from clear to white and reddish brown. Its hardness is 2.7, specific gravity 1.60, luster vitreous, and fracture conchoidal. Carnallite is highly hygroscopic, causing shipments containing this mineral to cake badly in transit or storage. It has a sharp, bitter taste.

#### ORIGIN AND MODE OF OCCURRENCE

The occurrences of potassium in nature are as varied as its uses. It is found in rock and mineral deposits and in the waters of lakes, rivers and the oceans. Clarke<sup>4</sup> estimates that 3.11 per cent of the earth's land surface is composed of potash and that the waters of the oceans contain nearly 0.05 per cent. Much potash occurs in forms that render it unusable at present. The most easily available and usable form of potash is the mineral sylvite. Sylvite deposits are believed to have resulted from the evaporation of saline waters under conditions of hot, arid climate, in an arm of a sea or a large saline inland lake. The salts, principally sodium chloride, thus selectively precipitated were covered by sedimentary rocks such as clays, limestones and sandstones.

Some producing potash mines, including those of southern Germany (Baden), Spain and southern Poland, are working potash deposits of Tertiary age, but many of the world's best known deposits, notably those of northern Germany, northern Poland, Russia and New Mexico, are of Permian age.

The extent of the Permian salt basin in the United States has not been clearly defined, but in general terms it includes parts of Colorado, Kansas, Oklahoma, Texas and New Mexico. As early as 1887 Hill<sup>5</sup> called attention to the enormous deposits of salt and gypsum in what was known as the Permian Basin, though not then fully accepted as such, and also to the fact that its deepest part was not far from its southern border. The salt series of the Permian Basin in New Mexico and Texas has thus far yielded no fossils,<sup>9</sup> however, the succession of

salts, though complex, indicates sea water as the primary source, by the succession of anhydrite, halite, and then the more soluble minerals in spongelike masses of halite. The salts from land-locked basins ordinarily include considerable amounts of calcium and magnesium carbonate, borates, and other minerals that are not found in saline residues from marine water. The repetition of beds of clay, anhydrite and halite indicates that the cycle of evaporation and deposition was interrupted and renewed many times.

In New Mexico, the sylvite has been found in important quantities only on the western flank of the basin, where it rises to an old erosion surface now buried some hundred feet by later sediments. These deposits may have been shifted by terrestrial movement while the potash was still in the mother liquors and tilted after they were precipitated from the brines. After the deposition of much of the salt series, mineralogic changes were induced by continued reactions of the salts with the mother liquors, as has been ably illustrated by Schaller and Henderson.<sup>17</sup> These authors noted in their core samples evidence of successive replacement of anhydrite or halite by polyhalite or sylvite and other similar changes.

The salt deposits in southeastern New Mexico are younger than those of central Kansas, where traces of polyhalite have been found, and record the gradual southwestward withdrawal of the Permian sea.

The southern part of the Permian Basin, in New Mexico and Texas, is a broad, asymmetric syncline trending generally northeast and characterized by gentle dips on the east and steeper dips on the west. The Delaware Basin, an arm of the Permian Basin in southeastern New Mexico and adjoining parts of Texas, is roughly circular in outline and within it occurs the greatest thickness of upper Permian rocks in the Southwest. The rim of the Delaware Basin is formed by the Capitan dolomitic limestone and apparently resulted chiefly from the combined effects of reef building and structural warping. Correlation studies in this part of the basin are especially difficult, even with samples of well cuttings, because of rapid changes in lithology, the alteration of outcropping beds on the rim of the basin, and the failure of some known subsurface beds to crop out.

In the central part of the Delaware Basin, in Lea County, New Mexico, about 1000 ft. of salt is found through a stratigraphic thickness of as much as 4000 ft., divisible into a lower salt series (Castile anhydrite) and an upper salt series (Salado halite). These saline beds, cut off by erosion in the valley of the Pecos River, thin out toward the other boundaries of the basin.

The Castile anhydrite, or lower salt series<sup>10</sup> consists of gray anhydrite, clean white rock salt, some sandstone and dolomitic limestone. Commercial amounts of potash have not been found in this series.



The Salado halite, or upper salt series, is made up of pinkish to white rock salt, reddish and shaly sandstones and massive anhydrite, which locally is replaced by polyhalite. The upper part of this series contains the local concentrations of the chloride and sulphate salts of potassium, such as occur about 20 miles east of Carlsbad.

Polyhalite has been found in the southern portion of the Permian Basin through an area of about 40,000 square miles.<sup>9</sup> Sylvite, carnallite

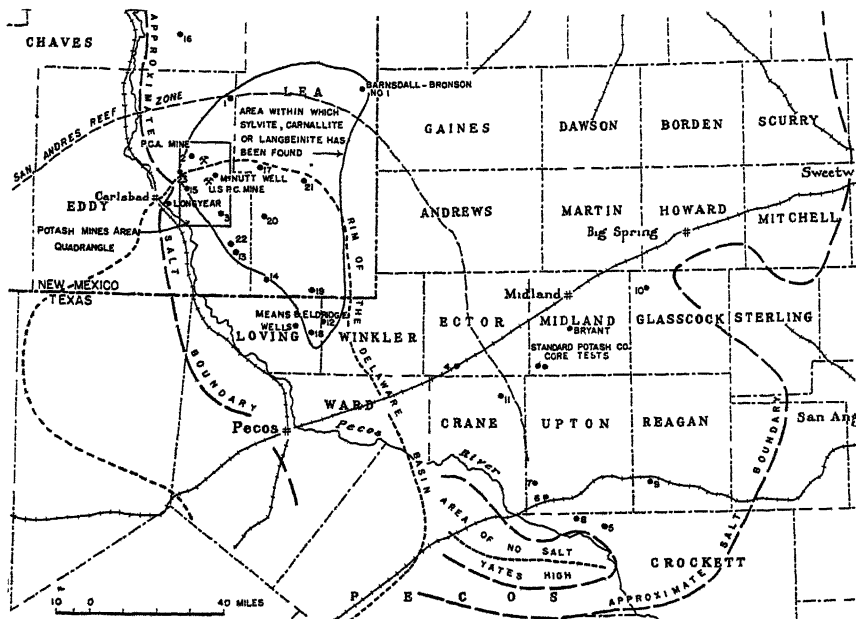


FIG. 1.—SOUTHERN END OF PERMIAN BASIN IN NEW MEXICO AND TEXAS.

Compiled after maps by Mansfield and Lang.<sup>8,10</sup> Shows the location of Longyear well,<sup>15</sup> where the first core test was made in the Permian Basin for potash exploration, 1917. Bryant well,<sup>19</sup> from which the first polyhalite in the Permian Basin was identified, 1921. Means well, drilled in 1921, is located near the thickest part of the salt series. It was offset by Eldridge core test,<sup>8</sup> the first well core drilled for potash in Texas, completed in 1926. McNutt well,<sup>11</sup> where sylvite was first discovered in the Permian Basin in 1925. Barnsdale-Bronson No. 1 well, the northernmost well showing soluble chlorides of potash. Standard Potash Company's two core tests.<sup>27</sup> Nos. 1 to 23, Government core tests drilled for potash in 1926-1931.<sup>10</sup>

or langbeinite have been identified in the cuttings from holes drilled for oil and in the cores of test holes drilled for potash in an area of 3000 square miles in southeastern New Mexico and the adjacent part of Texas (Fig. 1). It is reasonable to expect that this area may be extended or that other areas may be discovered as more information is obtained from drilling or from geophysical prospecting. This 3000 square mile area is bounded in part on the east by the Capitan reef, here extended beneath cover, and on the west by the eroded and covered edge of the Salado halite. One developed potash area is north and another south of the supposed location of the Capitan reef. Potash salts other than polyhalite have

also been found north of the San Andres reef zone (Fig. 1), which lies to the north of the Capitan reef and at some depth below the salt series.

The area intensively tested by core drilling is shown on the 15-minute topographic map of the U. S. Geological Survey covering the potash mines area extending 25 miles north and 15 miles east of Loving, N. M. The geology of the potash-bearing beds within this area is relatively simple as compared with that of the basin as a whole. The correlation of one hole with another is easy because of a persistent zone of white polyhalite occurring a few feet above a prominent anhydrite zone. This sequence occurs at two horizons in the southern part of the area and may cause some confusion without a study of the entire series. A correlation based on the McNutt No. 1 well supplemented by later drilling indicates 40 beds that show enrichment of potash.<sup>19</sup> Beginning at the top, beds 1 to 16 range from a high percentage of polyhalite to a high percentage of anhydrite. In beds 17 to 28 sylvite predominates except in bed 23, the distinctive white polyhalite bed, and bed 24, the anhydrite used in correlation. The potash mineral in beds 29 to 40 is polyhalite.

These beds are not uniform in lithology, thickness or intervals of succession. The so-called polyhalite beds vary from hole to hole in thickness and range from nearly pure polyhalite to anhydrite. The amount of halite in such beds is relatively low. The most promising core test of polyhalite showed one bed 9 ft. thick containing 13.5 per cent of  $K_2O$ . Bed 24, generally recognized as anhydrite throughout the field, locally contains high percentages of polyhalite. The sylvite beds vary similarly in the ratio of sylvite to halite. Carnallite is found in greatest abundance in bed 17, and langbeinite in beds 21 and 26. Beds 17, 18, 25, 26 and 28, as at present indefinitely outlined, each includes more than 4 ft. of sylvite, carnallite or langbeinite and more than 14 per cent of  $K_2O$  over an area of from 1 to more than 25 square miles. In the two developed areas bed 28 is worked. Kieserite, which abounds in Europe, is widely scattered in small quantities through the Salado halite. The most important deposit found in New Mexico is in T. 22 S., R. 30 E., New Mexico meridian. In an area a few miles to the south of the United States Potash Company's mine there are in addition to sylvite two beds high in langbeinite. To the northwest is a thick, rich sylvite deposit discovered by the Government and included in a Government reserve, subject to prior rights; to the north the Potash Company of America and to the east a deposit high in langbeinite.

Occasionally salt horses<sup>1</sup> occupying small areas are found in the two mines developed in New Mexico, similar to occurrences in European potash mines. Thus failure to find potash in a core through a particular bed is not conclusive evidence that rich salts may not be found within a short distance from the location of the hole.

Structure contours drawn on bed 23 show a gentle dip to the east with some folding; a troughlike depression extends northeastward, beginning some distance east of the United States Potash Company's mine, and some folding, which may reflect the location of the concealed Capitan reef, occurs near the mine of the Potash Company of America.

#### DISTRIBUTION OF DEPOSITS

*United States.*—Many sources of potash exist in the United States, and some of these were used during the World War. In 1918, the year of the greatest production during the war period, the brine deposits of Nebraska, Utah, and California furnished 73 per cent, kelp 9 per cent, molasses distillery waste 6 per cent, alunite 5 per cent, and cement and blast-furnace dusts, beet-sugar water, wood ashes, wool washings and other sources 7 per cent.

The only three of these wartime sources that have been consistent sources of potash since the war are the Searles Lake brines in California and cement dust and molasses distillery wastes in Maryland. Production from the brines of Searles Lake, started in 1916, has been for many years the principal source of potash produced in America. The successful production of potash from the brines in Searles Lake was made possible through extensive research involving fundamental scientific work.<sup>38</sup> The development in New Mexico resulted from a long period of search in the Permian Basin by State and Federal scientists, and the gradual development by these agencies of public interest in the potash possibilities.

The New Mexico potash salts and Searles Lake brines contain ample reserves for any potash needs in this country for many decades. The production from cement dust and molasses waste is an incidental product. The working of potash deposits of other kinds will depend largely on their feasibility as sources of aluminum, magnesium or other co-products. Efforts are being made to produce potash from western Utah brines, and research work is being done on alunite. Interest in other potential sources of potash is more or less dormant.

*World.*—The saline mineral deposits are the chief world sources of potash. Deposits of this kind are now being worked in Germany, France, Spain, Russia, Poland and New Mexico.

The principal foreign brine deposits are those of the Dead Sea, in Palestine, now being worked by the British and yielding both potash and bromine. A persistent effort has been made by Italy to produce potash from sea water, and some was produced from saline springs and brine wells in Ethiopia until 1930, from lands held under an Italian concession obtained in 1914. This Ethiopian potash was an important source to the Allies during the World War. Potassium nitrate is produced from the Chilean sodium nitrate deposits and also has long been a

product of India. Potash is obtained from kelp in the British and Japanese islands and small quantities of potash are obtained from various sources in many countries.

The German deposits nearly encircle the Harz, Flechtigen and Thüringer Wald uplifts, underlie the intervening basins, and extend northwest under the Hanoverian lowlands and southwestward into Thuringia, embracing a potential productive area that is thought to be as much as 24,000 square miles. The deposits worked may be divided into six districts—Werra-Fulda; South Harz (including Quesfurter and Mansfelder trough); North Hanover and North Harz; Magdeburg-Halberstadt-Stassfurt Basin; lower Rhine near Wesel; and the border district in Baden.

Potash was first discovered in Alsace (now in France) about 21 miles northwest of Mulhouse, in 1869, but the deposits now being worked were discovered as a result of drilling an exploratory hole for oil in 1904. The results of other holes drilled warranted development, and the first shaft was completed in 1909; 37,000 metric tons of crude salts were mined in 1910. According to Gale,<sup>15</sup> the deposit lies in a graben along the Rhine River about 25 miles north of the Swiss border and 375 miles by river from Rotterdam. Below the river alluvium are shale, rock salt, gypsum or anhydrite, and other deposits indicating deposition from standing evaporating water. The upper bed, 3 to 5 ft. thick, contains about 21 per cent of  $K_2O$  and covers about 84 sq. km. (32 sq. miles). The lower bed, 10 to 16 ft. thick, averaging 12 to 15 per cent  $K_2O$ , covers 172 sq. km. (66 sq. miles). The reserves are estimated in round figures at an equivalent of 300,000,000 tons  $K_2O$ .

The Spanish deposits lie about 60 km. from Barcelona, between the Pyrenees Mountains on the north and the Catalanian Ridge on the south, in an area about 75 miles long with a maximum width of 15 to 18 miles.<sup>6</sup> Three mines now in operation are often referred to as Suria, Cardona and Ibericas.

The Russian deposits near Solikamsk, where prospecting began in 1916 and development in 1925–1927, lie on the west side of the Ural Mountains, on the Ussolka River, a branch of the Volga, about 200 km. from Perm, on the Kama River, 1800 km. from Leningrad, and 2000 km. from the Black Sea. The deposits as explored extend about 30 km. north and south and 10 km. east and west. One mine is in the northern section in the vicinity of Solikamsk, and another is in the southern section in the vicinity of Berezniki. The production of crude salts from these deposits during 1935 is reported as 1,319,000 tons.

The Polish deposits, discovered in 1854 and first exploited in 1862, were worked but little prior to 1922. They lie north of the Carpathian Range, extending from Kalusz to Stebnik, a distance of 70 km., in a belt 3 to 4 km. wide. The three mines are at Kalusz, Holyn and Stebnik.

The potash reserves, except those of France, are difficult to estimate even approximately on account of slight prospecting by drilling and development and the irregularity in extent, thickness, and percentage of pure potash at any known horizon. Sufficient reserves are apparent in the principal producing countries to maintain operations from 100 to 1000 years, and long before these are exhausted others will be found by further exploration, or the reserves can be increased by including lower grade salts. For instance, in New Mexico, by lowering the average grade from 25 to 9 per cent, in terms of pure potash, the thickness of the deposits in the United States Potash Company's shafts can be increased from 10 to 140 feet.

#### POLITICAL AND COMMERCIAL CONTROL

The production of potash from saline mineral deposits was from its inception under varying degrees of political control. The Heydt and Manteuffel companion shafts, sunk near Stassfurt, for the production of rock salt by the Prussian Government, initiated the production of potash late in 1861. During 1856 and 1857, while these shafts were being sunk, several hundred tons of potash salts were thrown on the dump as worthless, but some sales were made for agricultural purposes. The Leopoldshall shaft, developed by the Duchy of Anhalt, began to produce potash in 1862, and for the next 13 years production was confined to those two state-developed properties. The first mineral mined was carnallite.

These two producers agreed on a price for crude salts, but the manufacturing branch of the industry, a private affair, increased the number of factories from three in 1862<sup>6</sup> to eighteen by 1865, and competition developed not only between the potash factories but between potash and soda ash. This competition resulted in a decline in prices for 80 per cent KCl from 320 marks (about \$76) a ton in 1863 to 149.60 marks in 1865, which caused five of the eighteen refineries to go bankrupt and others to change hands.<sup>22</sup> By 1869 the price rose to 175 marks, but in 1873 it reached a new low point of 120 marks. This resulted in financial casualties and the formation in 1876 of the first potash syndicate. This syndicate was little more than an association for weekly price agreements, and it seems to have been dissolved a year later. In 1878 the price of muriate was further reduced to 95 marks.

With the beginning of intensive commercial development, Prussia adopted a new mining law on June 24, 1865, soon followed by similar laws in most of the other states. This law recognized freedom in prospecting by anyone anywhere, on application to the surface owner for permission, and after discovery of potash the owner could be forced, on application to the district mining official, to grant a concession for exploitation.

The first private mine was the Douglashall, sunk near Westeregeln. Here two shafts were completed in 1873 and 1874, and operations were begun in 1875, followed by the Salzbergwerks in 1877.

The long-established idea of state control of industry led the Prussian Fisc in 1877 to endeavor to unite the four mining enterprises under a working agreement for allocation of output and regulation of prices, but the two private operators refused to accept the proposed terms, which would have reduced their output.

An agreement was reached in 1879, marking the first potash cartel, which in effect limited the output of carnallite for five years, determined its distribution to the refineries, and contained price provisions. The kainite producers entered into a similar agreement. The muriate factories fixed the prices for the concentrated products and in 1883 formed an association for marketing such products through a central sales office. From that time to 1910 the cartel was a rather loose organization depending on term agreements. On June 30, 1909, the last day of the agreement then in force, a discord existed between those wishing to maintain prices and those promulgating the policy of lower prices and larger sales. As no agreement had been reached by midnight, all were free to make individual contracts, and within a few hours the Schmidtman and Westeregeln group made with American agents extensive contracts involving the equivalent of 120,000 tons of  $K_2O$  annually, at prices ranging from 30 to 40 per cent below the syndicate price on two-year contracts, with options on a five-year extension. After considerable international negotiations, Germany finally, on May 25, 1910, enacted a law which obligated the Americans to surrender their contracts, at a loss of \$25,000,000 on the contracts, and to pay \$2,000,000 to \$3,000,000 in surtaxes to the German Government.<sup>6</sup> The Schmidtman prices at that time show the prices at which, it is believed, several German mines could then produce potash at a profit. The Schmidtman and syndicate prices per metric ton for the several grades of salt, c.i.f. Atlantic ports, were respectively as follows: Muriate, \$20.40, \$32.98; sulphate, \$25.80, \$38.80; double manure salts, \$14.14, \$20.37; 20 per cent manure salts, \$7.13, \$11.64; hartsalz, \$5.72, \$8.73; kainite, \$5.16, \$7.03.

The law of 1910, intended to control production in Germany through allotments, promoted conditions it was intended to eliminate and resulted in an orgy of sinking new shafts and the creation of several new concerns or subsidiaries by establishing compacts to protect the allotments formerly enjoyed. By April 1911 the 69 syndicate mines were being increased by 79, and two years later, in the spring of 1913, there were 127 mines operating and 132 under development, a total of 259 shafts.<sup>6</sup> More than 200 of these mines have since been closed, and the consumers of potash are paying for this folly of enacting laws that aggravated rather

than remedied a bad economic situation. This interference with normal development materially added to the price the consumer paid for potash, and no small part of this increase has been paid by American buyers. It is safe to assume that the United States Government, through its expenditure of approximately \$2,000,000 in 20 years in the successful search for potash in this country, has caused an annual saving of half that amount to the American consumer. This search has achieved American independence in potash and has established a new industry of material benefit to American labor and transportation companies.

The control of the German industry by allotments and price fixing, the publicity incident thereto, and the inability to get potash during the World War caused the countries dependent on German potash to seek domestic sources of supply. Now the control of the world supply of potash is shared with Germany by France and Poland through national boundary adjustments; by Russia, Spain, and the United States through development of deposits; by Palestine, Italy, and Ethiopia through production from brines; and by the British Isles and Japan through production from kelp. All the producers that have surpluses for export, except those in the United States, are now reported to be in agreement with the syndicate on world markets and prices.

The potash industry in New Mexico is under political control to the extent that the present known deposits, largely on State and Federal lands, are operated under leases in accordance with the laws and regulations of the State and Government. There is no political control over production in California or Maryland except the State and Federal laws applicable to industry in general.

To prevent a duplication in the United States of the overdevelopment in Germany, the Secretary of the Interior has ceased to issue permits to prospect for potash on public lands or to issue leases other than those mandatory by laws as a result of discovery on or under a pre-existing prospecting permit. At present two concerns are producing potash from Government lands, and plans are under way for a third operation to begin production some time in 1938.

#### PRODUCTION AND CONSUMPTION

*United States.*—The initial American production of potash from mineral sources of 1090 short tons of pure potash in 1915 was increased in 1918 to 54,803 tons or about 20 per cent of the normal American requirements; of this quantity 53 per cent came from the shallow lakes in western Nebraska, 34 per cent from California, 7 per cent from Utah, and 6 per cent from other sources. With imports available from Germany in 1921 the American production dropped to 10,171 tons, but it gradually increased to 61,270 tons in 1930, most of which came from California. By the addition of the New Mexico development and

increased capacity in California, the American output was more than trebled during the depression years, reaching 192,793 short tons of pure potash in 1935, as compared with 228,556 tons imported for fertilizer purpose and 12,954 tons imported for industrial use during the same year. The imports, exports, consumption and average value of potash are shown graphically in Fig. 2.

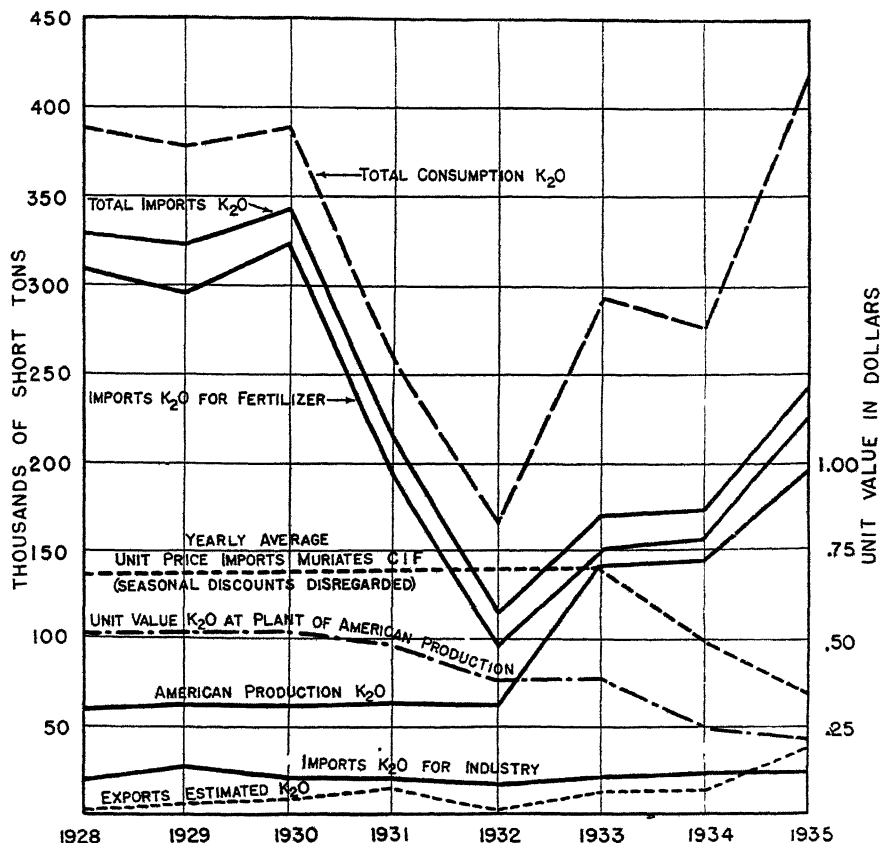


FIG. 2.—TREND IN AMERICAN PRODUCTION, IMPORTS, EXPORTS, APPARENT CONSUMPTION AND AVERAGE VALUE OF AMERICAN POTASH, 1928-1935.

Figures on right side refer only to unit values of imports and domestic production.

The total hoisting capacity of the two mines in New Mexico is now 350 tons of crude salts an hour, an amount sufficient to supply all domestic needs with a surplus for export. The two refineries in New Mexico and one in California have a capacity to produce 425,000 short tons of potassium chloride annually. The total production in 1935 of crude salts from the two mines in New Mexico, of refined salts in California, and the small tonnage in Maryland was about 600,000 tons, containing the



equivalent of 192,793 short tons of pure potash. The exports in 1935 amounted to 75,985 short tons for agricultural use and 3641 tons for chemical purposes.

The imports of potash for consumption in agriculture show a trend to change from lower grade to the higher grade salts, no doubt due to the competition of high-grade muriate produced in America. The imports of kainite dropped from a peak of 640,042 short tons in 1910 to 82,888 tons in 1935, and manure salts from 453,242 tons in 1928 to 95,563 tons in 1935. Imported muriate reached a peak of 306,047 tons in 1930 as compared with 87,761 tons in 1932 and 262,370 in 1935. By adding the American production the apparent consumption of muriate for 1935 was about one-third greater than in 1930, the year of maximum imports of pure potash for fertilizer use.

In 1933 the imports of potash salts chiefly for chemical uses amounted to 53,858 short tons, valued at \$3,465,030. They decreased to 34,176 tons in 1935, when only five items exceeded 1000 tons—bitartrate (argols), 8184 tons; carbonate, 2063 tons; caustic, 1713 tons; chlorate and perchlorate, 7159 tons, and crude nitrate 13,627 tons.

*World.*—Alunite was mined for the production of alum in Italy as early as 1816.<sup>3</sup> The initial production of soluble potash salts was made in Germany in 1861. In Poland potash salts were noted at Kalusz in 1854; the mine was leased but it did not prosper. Production has been continuous since 1921. The first production of soluble salts in Alsace was in 1910, in Spain in 1925, in Russia in 1930 and in the United States in 1931. Production from brines was started in the United States and Ethiopia in 1915 and in Palestine in 1930.

The statistics on world deliveries of pure potash are somewhat contradictory, but during several recent years the annual production has exceeded 2,000,000 metric tons, or about twice the amount delivered in 1913. In 1935 the Polish output, in terms of metric tons of pure potash, was 71,539 tons; Spanish, 121,372 tons; French, 347,300 tons; German, 1,830,000 tons, an all-time record; United States 174,897 tons. Russian production is estimated at 130,000 tons.

From the initial production in Germany to 1879 the German exports for industry exceeded that for agriculture, and to 1890 the consumption in Germany for industry exceeded that of agriculture. In recent years 90 per cent of the potash produced has been used in fertilizers.

Potash produced and sold in the United States is reported in terms of merchantable salts, which range in purity from the 20 per cent by-product salts produced in Maryland to potassium chloride of a purity suitable for the chemical trade. There are no published figures for the mine production in New Mexico, the refinery output in California or for the Maryland output, for the reason that there are fewer than three producers in any of these states.

The statistics on world production imports and exports is published by the Bureau of Mines. The production of potassium compounds made in America is published biennially by the Bureau of the Census, but these statistics for 1933 are limited to the output of potassium acetate, bitartrate, citrate hydroxide and iodide. Other compounds, such as the nitrate, if any, and carbonate, are consolidated under a single item, as the Bureau does not reveal production of any compound if there are fewer than three producers.

Caution is necessary in comparing the statistical data on the value of potash fertilizer salts produced in America with the value of salts imported or exported, as all sales of potash salts of agricultural grade are made at the same price quotations for the respective grades c.i.f. Atlantic, Gulf and Pacific ports, and to these prices freight to interior delivery points is added. Potash imported into America in 1934 was valued at an average of \$50.21 a ton of pure potash at the seaboard. The average amount received for the domestic output at the points of production, such as Carlsbad, N. M., and Trona, Calif., in 1934 was \$24.65 a ton, the difference being largely a matter of land and water freight charges to Atlantic seaboard ports from the mines. Salts for export brought an apparent higher price than either imported or domestic salts, as the selling price included the freight charges to the nearest port and in some sales may have included freight to foreign countries. The  $K_2O$  content of salts is not revealed, but on the assumption that it averaged 50 per cent  $K_2O$  the pure potash exported was valued at \$65.62 a ton.

#### EXPLORATION AND PROSPECTING

*Search for Potash.*—The potash salts of commerce come largely from deep bedded saline deposits. These deposits were first discovered in the course of mining salt or drilling for salt, oil or water. After the presence of potash has been established, the extent and value of the deposit are determined by core tests. In Germany one core test at least was considered necessary before starting shaft sinking. In Alsace the entire deposit was well defined during the early stages of development by the drilling of 165 core tests. Early explorations in the United States by Government agencies and others naturally led to the investigation of ancient lake brines and salts in the western areas favorable to brine accumulations, such as ancient Lake Bonneville, of which Salt Lake, Utah, is a remnant; ancient Lake Lahontan, of which Pyramid Lake, Nev., is a remnant; the Owens Valley region, California, in which Searles Lake is located; the Salton Sea area, in southern California, which was cut off from the Gulf of California by the delta of the Colorado River; and the Permian Basin of New Mexico and Texas. Investigations were also made of the shallow lake regions of western Nebraska, and the Permian Basin, and of the eastern salt deposits, including salt beds cut

in drilling for oil. Still other investigations were made of rocks containing potash, such as feldspar, alunite, greensands, leucite and shales, of the dust from cement and blast-furnace operations, and of organic substances.

The discovery and development of the potash deposits in the Permian salt beds progressed slowly, owing to the war and to the lack of drilling for oil and of interest in potash possibilities by those responsible for the drilling. However, the sequence of events is somewhat as follows:

In 1911 Phalen<sup>13</sup> pointed out that the red beds of the southwestern part of the United States contain deposits of gypsum and salt and were worth notice as possible sources of potash salts. In 1912 J. A. Udden, then director of the Texas Bureau of Economic Geology, found potash in the brine of the Spurr well, drilled for water in Dickens County, Tex.;<sup>27</sup> in 1915 he found some red salts with 9.2 per cent  $K_2O$  in the Boden well, in Potter County, and red crystals with 6.1 per cent and clear crystals with 10.5 per cent  $K_2O$  in the Miller well, in Randall County. The salt lake southeast of Carlsbad, N. M., now used for impounding the waste salts from the refinery of the United States Potash Co., was examined for potash in 1912 with negative results.

Although polyhalite and other minerals had been previously mentioned and suspected, the first definite identification of polyhalite was made by R. K. Bailey in the laboratory of the United States Geological Survey early in 1921, in cuttings from the Bryant well, in Midland County, Tex., and about the same time he discovered polyhalite in the cuttings of the Burns well, in Dawson County. Two specimens weighing about one ounce each were found in the cuttings of the Pitts Oil Company's well, in Ward County, this same year. The Means well, in Loving County, was the first in which samples of cuttings were taken at 5-ft. intervals and carefully analyzed. Later it was offset by the Eldridge core test, the first in Texas. A dozen or more beds were found with relatively pure polyhalite or some potash enrichment. The McDowell well, in Glasscock County, although not so completely sampled, disclosed seven potash-bearing beds. The Jones well, in Borden County, drilled about the same time, also contained some potash.

In 1917, prior to the identification of polyhalite in Texas, two wells were drilled specifically for potash in the Permian Basin—one churn-drilled by the United States Geological Survey at Cliffside in Potter County, Tex., between the Boden and Miller wells, in the area which up to that time had showed the most promising potash possibilities, and the other, a core test drilled by E. J. Longyear, near Carlsbad, N. M., about 15 miles west of the present developed area. Neither of these wells revealed any notable amount of potash. From 1922 to 1925 the cuttings of many drill holes were examined, and an area in which polyhalite could normally be expected was outlined. In 1925, however, a rather high percentage of potash was found by V. H. McNutt in poly-

halite at several horizons in the Snowden-McSweeney-McNutt oil test in Eddy County, N. M. The cuttings from this well were later carefully examined petrographically by the United States Geological Survey and independently on the initiative of McNutt, with the gratifying discovery of sylvite in a zone which by chemical analysis yielded only 3.09 per cent  $K_2O$ . The potential significance of this small percentage was readily appraised by McNutt, with the result that core testing was started, and in the first hole sylvite was discovered equal in richness to some crude salts being mined in Europe.

*Prospecting.*—Under the provisions of the act of June 25, 1926, drilling for potash in the Permian Basin was pursued by the Government through a cooperative arrangement between the Bureau of Mines and the Geological Survey. During the next five years 23 holes were drilled by the Government in the basin. The discoveries resulting from the drilling of the 23 core tests by the Government and the 71 private core tests and the examination of the cuttings by the Government from holes drilled for oil contributed materially to outlining the part of the Permian Basin favorable for potash explorations.

The solution used in drilling is important in prospecting soluble formations. In the Longyear hole, mentioned above, both oil and brine solutions were tried. In later drilling, when it was found that a saturated salt brine was inadequate, 3 lb. of magnesium chloride was added per gallon of saturated salt solution; later  $\frac{1}{4}$  lb. of potassium chloride was added to prevent loss of core through dissolving by the solution. Finally a saturated solution of crude mine salts was used. The salts contained about 40 per cent sylvite and 60 per cent halite. As much magnesium chloride was then added as was required for saturation at bottom temperature in the hole. The test of saturation was to drop the temperature one degree and note whether any precipitate formed. If no precipitation occurred it was evidence of lack of saturation. As a result a core of the entire formation is now normally obtained, and there is little or no etching of the core by the drilling solution.

Double core barrels, which permit the inner barrel to remain stationary while the outer barrel rotates, are universally used in the Permian Basin. Diamonds of various types were tried, but those with a sharp cutting edge were the most satisfactory. Bort is also used. Stellite bits were found satisfactory, but some redesigning was required to keep the circulating solution close to the cutting edge. The 15-ft. core barrel is usually pulled after cutting about 12 ft. of formation. The diameter of the core is  $2\frac{1}{2}$  inches.

In normal practice a hole is churn-drilled to the top of the salt. The water encountered in drilling is cemented off, and coring is started in the salt formation. Recent core drilling has been done at the rate of about 40 ft. a day. The contract price has ranged from \$4 to \$17 a foot. The

price was affected by changing economic conditions, the contracting of one or more holes at a time, the length of moves, the cost of road building to the location, and the amount of coring, which ranged from 100 to 1676 ft. Before the local salts were used the cost of the drilling solution and the possibility of losing it in the formation were important items in determining the contract price. Of the 94 core tests drilled for potash in the Permian Basin 81 have been drilled in New Mexico and 13 in Texas.

*Core Sampling.*—In European prospecting the general practice is to drill cores 5 to 8 in. in diameter, using a single core barrel. Sampling is done by drilling a hole about  $\frac{5}{8}$  in. in diameter longitudinally through the center of the core. In this way a sample is obtained that is unaffected by the selective dissolving on the outside of the core.

In New Mexico the sections of core taken from the core barrel are placed in a single trough. It is then logged, transferred to a core box, and sent to the laboratory for mineralogic examination and sampling. The entire core is examined mineralogically and samples for analysis are cut either from the entire length or only from the portions that are of potential economic value or that occur at the same horizons as valuable beds in near-by holes. Sections of the core containing polyhalite and langbeinite are split in half with a regular core splitter. One half is preserved for a permanent record, and the other half is split again. One quarter is either ground or a portion is taken for analysis, and the other quarter, if the hole is on Government land, is sent to the Geological Survey for mineralogic and chemical check. Core containing the softer minerals, sylvite, halite and carnallite, is cut with an ordinary hand saw or band saw, and the sawdust from each cut is collected separately and analyzed. Most of the core tests so far made have been on Government land. In the Geological Survey laboratory the sample is ground from the center or inside portion of the core with an emery wheel, the amount taken being proportional to the length of each piece of core sampled. A small amount of the ground salt is crushed to a powder, immersed in oil with known index of refraction, and examined mineralogically under a microscope. Analyses by the platinic chloride method are made by the Government and one of the producers in New Mexico, and the cobaltic nitrate method is used by another producer. A volumetric method has been used to great advantage in determining the percentage of potash at the drilling site where prompt results were required.

#### MINING METHODS

*America.*—The first shaft for potash in New Mexico was started near Carlsbad by the United States Potash Co. in December 1929 and completed in January 1931. In 1933 the air shaft was completed by this company. The Potash Company of America started and completed its

first shaft in 1933. Its air shaft, started in 1934, and intermittently sunk was completed in November 1936.

In sinking,<sup>21</sup> jackhammers with straight cruciform bits and the usual V-type cut of shaft round were tried originally. However, it was found that in many of the strata, particularly in clays and sands, the shots would chamber and not break clean. This difficulty was overcome by the use of jackhammers with nonrotating chisel bits, similar to those employed in breaking up street pavements. These bits were entirely satisfactory in soft formations and eliminated the necessity of blasting. Regular rounds and blasting are now used only in sinking through sandstone, limestone, anhydrite and salt; 32 holes are used in each round in limestone and anhydrite, and 52 in the salt sections. A record of sinking 296 ft. and cutting four stations was made for one month.

The potash salts are mined by the room-and-pillar method, as in coal mining. Normally the salts in 50 per cent of the area are recovered in the first mining, and it is believed that 90 per cent of the potash can be obtained without back filling.

Potash salts are hard to break with an advancing face. The ore is not hard, but is tough and tenacious. When blasted it tends to break in slabs rather than in fine pieces. This behavior decreases the mechanical loading efficiency. Breaking the salt into small lumps by an excess of explosions gives a decided advantage in handling. At first all drilling was done by compressed-air machines, different makes being tried, and blasting was done from the solid by means of fuse and cap. As an aid in blasting and loading, all faces are now undercut by short-wall chain machines. There are several advantages in undercutting, such as a saving in the cost and handling of explosives, a smooth surface upon which the mechanical loading units can operate with increased efficiency, and better control in breaking to the bottom of the deposit without leaving high-grade salts or including salt of no value.

The cutter bars of the undercutters range from  $7\frac{1}{2}$  to  $8\frac{1}{2}$  ft. in length and operate at a speed of 4 to 6 in. a minute. Cutting bits of several types have been tried and hard-surfaced bits are now used. Those pointed with stellite or other alloy were good for about 80 to 100 linear feet.

Mounted electric auger drills operated by a  $1\frac{1}{2}$ -hp. motor are used in drilling the holes for blasting in the thick salts.<sup>14</sup> The holes are  $1\frac{5}{8}$  to  $1\frac{7}{8}$  in. in diameter, have an average depth of  $8\frac{1}{2}$  ft., and are drilled at a rate of 32 in. a minute. Five rows of holes are spaced vertically and about 14 horizontally in a face 10 ft. high and 40 ft. wide. At one mine all shots are fired electrically from a control station and delay detonators are used to get the proper rotation of shots. Few misfires occur.

Four types of loader machines are used—for a minimum headroom of about 8 ft., a three-drum Sullivan scraper slide with a box-type scraper and a Goodman electrohydraulic shovel of the caterpillar type; for 6

ft. or less of headroom, a Sullivan scraper loader designed to load in rooms 60 ft. wide and 200 ft. long, and a Jeffrey face loader with an 11-ft. loading boom. Both storage-battery locomotives and trolley locomotives are used. The cars range from  $4\frac{1}{2}$ -ton side-dump Granby cars to 5-ton solid cars for dumping in Card rotary dumps. The salts are dumped underground into skip pockets and hoisted in 5-ton underslung skips.

At both mines the loading of the skips is semiautomatic. At one the weight is the controlling factor; at the other, when the ore fills the skip it operates a trip that shuts off the flow. At one mine the salt is crushed to 3 in. underground, and at the other the salt as mined is passed through a grizzly and hoisted to be crushed on the surface.

The double-cylinder drum hoists are operated electrically, with the rope speeds 1000 and 1200 ft. a minute. The combined present hoisting capacity at the two mines is 350 tons an hour. Both hoists are equipped with electric speed and overwind controls. The headframes are 110 and 125 ft. high and are equipped with storage bins with a capacity for about 2-hr. run of the mine. The surface machine and electric shops are supplemented by underground shops in both mines.

Only traces of methane have been found in the analyses of mine air, but precautions are taken against any sudden liberation of flammable gas, such as sometimes occurs in European potash mines. A pocket of gas under high pressure was struck in sinking the first shaft. This gas contained hydrogen, methane and a high percentage of nitrogen. For ventilation, one company uses a fan with backward-curving blade, having a capacity of about 85,000 cu. ft. per minute. The other company has installed a 7 by  $3\frac{1}{2}$  ft. fan with a capacity of 150,000 cu. ft. a minute. Both are electrically operated, and housed in steel and concrete buildings so arranged that the air current can easily and quickly be reversed.

Concrete overcasts and undercasts are used in both mines to control ventilation. Some stoppings are built with salt moistened with a saturated brine, which causes it to become solid without dissolving out the small particles of salt and making it porous.

Ground temperatures in the salt seldom exceed 74° F., and air temperatures are rarely over 79° F., even on return air and in dead ends. Higher temperatures are expected as mining continues and subsidence begins.

*Europe.*—The potash-bearing beds in Europe vary considerably from those that are highly and complexly folded to those that are relatively flat, and the methods of mining necessarily vary accordingly. Rice<sup>16</sup> states that the method now generally employed in mining the steeply dipping deposits in German potash beds is overhand chamber stoping combined with back filling and pillars. After a stope has been opened the first slice from the roof is drilled and shot progressively over its entire area. Then only sufficient salt is drawn to keep about  $6\frac{1}{2}$  ft. of head-

room until a higher crosscut about 30 to 36 ft. up is reached. The stope is then emptied and filled with waste salt dumped from above. The filling is often obtained from a stope driven for that purpose supplemented by waste salt from the refinery, which, being moist, tends to solidify to a hard mass in the mined-out area. After the stope is filled within about 6 ft. of the top, the same procedure is followed in removing the next higher section. Room-and-pillar methods prevail in the flatter beds. In the French mines, where the beds have gentle dips, the potash is mined by a longwall advancing system, blocks of waste salt picked out during the course of mining being used for building pack walls behind which the finer waste salts are shoveled. The moist refinery wastes brought in from the surface, when placed in the pack walls, make the mass set almost like cement. Where waste salts are not readily available the pack walls are built at right angles to the face, and only half of the area is back-filled. In Poland the mined area is back-filled hydraulically with the waste from the refinery.

#### PREPARATION FOR MARKET<sup>21</sup>

The mechanical treatment of the crude salts as mined in New Mexico is similar in both plants, except that the Potash Company of America does primary crushing underground. The mine-run material is taken from the headframe bins by belt conveyers to crushing-plant bins and thence over crushers and screens of various types to make a 6 to 10-mesh product, sold to the trade as manure salts and containing from 40 to 48 per cent of sylvite ( $\text{KCl}$ ), equivalent to 25 to 30 per cent of  $\text{K}_2\text{O}$ . No hand picking or sorting is done in either plant, and the grades stated approximate the run-of-mine product.

At the United States Potash Company's refinery the crude crushed salts are converted by a process of solution and fractional crystallization, in a single refining step to a finished product averaging more than 99 per cent  $\text{KCl}$ . The process is based on the relative solubilities of sodium chloride and potassium chloride. When both salts are present in quantities sufficient to allow complete saturation they are radically different in their response to changes in temperature. Increase in temperature enhances the capacity of mother liquors for potassium chloride but effects little change in their capacity for sodium chloride. A selective extraction of potassium chloride is thus made possible. The process includes extraction, cooling, filtering and drying. Heat is introduced into the system by means of heat exchangers, fed with cold mother liquors. The process is cyclical in nature and largely continuous.

In producing the high-grade muriate the crude salts to be treated are dumped into a hopper near the plant and fed onto an inclined belt conveyor, which carries them to a surge bin in the plant. From this bin they pass over Traylor vibrating feeders to an elevator that discharges into



measuring bins, from which they go to a battery of dissolvers or extractors. The dissolvers when charged are closed, and preheated mother liquor is added to the salts to leach out the sylvite. The mother liquor, which has been previously saturated with sylvite and halite at room temperature and then preheated nearly to the boiling point, gives an increased potential capacity for sylvite of approximately 1 lb. of sylvite per gallon of mother liquor. This causes a selective action, dissolving the sylvite and leaving the salt (NaCl) behind. The residual salt is flushed from the dissolvers with cold water. The dissolvers are then recharged as outlined above, and the cycle is repeated.

The saturated liquor from the dissolvers is pumped continuously to three vacuum coolers in series. In these vessels constant boiling is maintained in three steps at progressively reduced temperatures. Steam from the liquor is condensed in tube condensers, and the residual gases are removed by ejectors. The saturated liquors from the dissolvers are finally reduced approximately to atmospheric or room temperature and, being charged with muriate (KCl) in suspension, are then fed onto a top-feed Oliver filter. Here the muriate is recovered, and the cooled mother liquor is returned to the head of the plant, where it is again heated and charged into the dissolvers. The muriate crystals are dried and screened and are then ready for shipment either in bulk or in bags. The refined muriate is clear white and has the physical appearance of table salt.

The refinery of the United States Potash Co., with a daily capacity of 200 tons of refined muriate (KCl), was placed in operation in September 1932, its capacity was doubled in 1934 and stepped up to 500 tons in 1936. The scheme of fractional crystallization to purify crude salts containing sylvite is not new, but improvements in apparatus, made available by pioneering American manufacturers of such equipment, have allowed prompt advantage to be taken of the latest technical advances. Alloys of various kinds, concrete and wood are used in appropriate places.

Steam for both power and heating is generated in Stirling boilers by the use of natural gas from eastern New Mexico. Turbogenerators of the Curtis type are used for power, and full use is made of exhaust steam from the turbines for heating. All condensates are returned to the boilers, to ensure a suitable boiler-feed water. Electrical energy is generated at 440 volts and is stepped up to 33,000 volts for transmission to the mine, about 13 miles to the north.

At the plant of the Potash Company of America the crude salts are concentrated by flotation. The separation by flotation of sylvite and halite, which differ only 0.16 in specific gravity, can be accomplished in the laboratory in different ways. Initial experimental work on mechanical separation of halite and sylvite by the United States Bureau of Mines led to cooperative experiments with the Potash Company of America at

the Missouri School of Mines, where small-scale tabling tests were made to produce a coarse-ground product, which was considered desirable at that time. Though these tests were favorable, it was decided to make further experiments before attempting production on a commercial scale.

Later research by the company's staff, independent of the Bureau of Mines' work on flotation, resulted in the development of the process now used at the company's plant. The plant has a daily capacity of 200 tons of muriate and work is under way to double this output. The adaptation of flotation to the salines is an outstanding development in milling operations.

In producing the refined product the crude salts from the crushing plant are transported over belt conveyers to two bins at the head of the concentration plant or refinery. The mill feed from the two storage bins goes into two pebble ball mills, to which is added brine saturated with sylvite and halite at toom temperature. This is done in order to preclude the solution of sylvite during grinding. The finely ground product from these mills goes into two Akins classifiers in closed circuit with the pebble mills, and the fines from the classifiers pass on to a Dorr thickener. From the thickener the pulp goes to a Minerals Separation Co. 12-cell rougher flotation machine, where reagents are added. At present the salt (halite) is floated and the sylvite depressed, but should it be desirable this process can be reversed.

The float or salt from the rougher is joined by the salt from the cleaner for further treatment to extract the residual sylvite. The salt tailings as discharged contain only a minimum of sylvite. The middlings from the first flotation machine or rougher go to a 6-cell cleaner for further treatment. The middlings from the cleaner are reground and re-treated, and the salt tailings from it join with the rougher salt as just mentioned.

The concentrated sylvite from both machines is taken to three Fletcher centrifuges, and the pulp from them to a Ruggles-Cole drier. The dried sylvite goes over a water-cooled drag conveyer to the warehouse for shipment or storage. This refined product, though retaining the pink color of the crude potash salts of this district, contains from 96 to more than 99 per cent of KCl.

At this mine all power is developed by three Diesel engines, with a total of 1800 hp. In addition there are three air compressors, two Diesel-driven and one motor-driven. The soft water for use in the jackets is shipped to the mine in railroad cars.

Very little water is used in the process at the mine of the Potash Company of America, that pumped from the shafts being sufficient, as the salts are not put into solution. An interesting development has been the use of the jacket water from the Diesel power plant to heat the brine developed in the process, this procedure having a desired cooling effect upon the jacket water, as well as affording a heat transfer. Water for

the United States Potash Company's refinery is obtained from the Pecos River, and some of it is softened for refinery use.

#### TESTS AND SPECIFICATIONS

For agricultural use the composition of the salts appears to be of little concern except for the soluble  $K_2O$  content, which is closely checked and regulated under the laws of the various states. The official and tentative methods of analyses of the Association of Official Agricultural Chemists govern to a wide extent in the matter of analytical methods used in analyzing potash for agricultural use and many of the concerns dealing with potash analyses receive periodical samples for check analyses. A petrographic check of the materials constituting a mixed fertilizer may be even more important than exact analyses of the fertilizer materials.

When mixed fertilizers are sold in bags, each bag must be labeled as to its content of the three fertilizer elements—nitrogen, phosphorus, and potash—and heavy fines are imposed for misrepresentation. To mix the fertilizer materials containing the three fertilizer elements with the 80 to 85 per cent of filler going into the mixture requires much skill on account of differences in the specific gravity, texture and granulation of the materials used.

The purchaser of fertilizer can depend on the percentages indicated on the tags, but this is insufficient to indicate the effect of fertilizers on the growing of crops. For instance, as shown by Turrentine,<sup>26</sup> salts marketed as high-grade kainite may not contain any of the mineral kainite, magnesium or potassium sulphates but be composed largely of potassium and sodium chloride, with the rock salt dominating. In using such so-called kainite the rock salt, even though at times beneficial, may counteract the benefits of the potassium content of the fertilizer. The expense of making tests on plants and the many years required for a series of plant experiments warrants a full analytical and mineralogical disclosure of the constituents used in the fertilizer and if not known a petrographic and analytical examination of the materials should be made and published with the experimental data.

Potassium chloride is the base used in America for the manufacture of chemicals for ceramic and other industrial uses. For ceramic uses the specifications are very rigid for elements, such as iron, that affect the color or elements that affect the texture, but considerable leeway is given for silica or aluminum and such elements as normally enter the mixture.

#### USES

The early settlers, on arriving in America, found the Indians using wood ashes and fish to fertilize their crops, and wood ashes continued to be the principal source of potash until some time after 1872, the year in which mineral potash was experimentally introduced to American agri-

culturists. About this same time the need of proper labels and guarantees for fertilizer was being advocated, and they were gradually adopted successively by the various states.

The chief use of potash until about 1890 was in dyeing and tanning, and the making of glass, pottery, soap, matches and explosives. Now about 90 per cent is used in agriculture and 10 per cent in industry.

Ample potash for plant growth occurs in most soils except in very sandy soils, peat and muck, but much of this potash is combined with silicates that are not sufficiently soluble to make the potash available in the quantities required. Enough of this potash becomes available each year to support a moderate plant growth, especially if the plant dies and decomposes in place, but under intensive cultivation, where a large part of the crop is removed, soluble potash must be added. Leaching, erosion, fixation, and removal of crops reduce the available potash.

One of the prime functions of potash is to aid in the formation and storage in the plant of starches and sugars. In addition to using potash to promote plant growth, more attention is now being given to improving the quality of the crop. Potash appears to build up and strengthen the cell walls of the straw in grains and of stalks in other plants. Plants well supplied with potash are more vigorous and resistant to disease, and apparently offer a greater resistance to the penetration of the cell walls and destruction by pests.

Potash is most effective when used in conjunction with proper amounts of other plant nutrients, such as phosphates and nitrates. Calcium, magnesium, sulphur and other elements are often beneficial. Potash adds a noticeable improvement in perfection of shape, flavor, color, and shipping and keeping qualities, thus fulfilling a very important function in vegetables and fruits, such as tomatoes, strawberries and apples, which are shipped long distances and held in cold storage for postseason markets. These qualities add more to the value of many products than an increase in volume of output. The lack of potash during the World War was regarded as the main cause of the bad condition in which the bulk of the vegetables reached the markets. Considerable emphasis has been placed on "potash hunger" and "potash starvation."

Annual statistics are not available on the American manufacture of potash compounds used in industry but some statistical data on American production are given in the reports of the United States Census of Manufacturers.<sup>2</sup> The largest item in the imports<sup>12</sup> of potash salts other than those used chiefly for fertilizer is potassium nitrate ( $\text{KNO}_3$ ). The imports of potassium nitrate, crude and refined, in terms of  $\text{K}_2\text{O}$ , amounted in 1935 to 5794 short tons. Chilean nitrate was long the source of the greater part of the raw material for this product. It is now finding competition in the manufacture of potassium nitrate from crude potash salts. Crude nitrate of potash is used in increasing quantities in fer-

tilizers, and refined nitrate is used for curing meats, in the manufacture of black powder, fireworks and tobacco products, and, to a small extent, in the ceramic industry and in drugs.

Potassium carbonate ( $K_2CO_3$ ) and bicarbonate ( $KHCO_3$ ), formerly obtained almost entirely from wood ashes, can now be produced in many ways, of which the Le Blanc process has been an important one. In this process the potash salts are mixed with powdered coal and limestone and heated in a furnace that converts them to carbonate, which in turn is purified by solution and evaporation. The magnesium-potash process, used extensively in Germany, depends on the decomposition of potassium chloride by carbon dioxide in a suspension of magnesium carbonate at a temperature below  $24^\circ C.$ , with the formation of the sparingly soluble salt  $KHCO_3 \cdot MgCO_3 \cdot 4H_2O$ , which is transformed into potassium carbonate and magnesium carbonate by treatment with hot water containing magnesium hydroxide. The impurities are in large part removed by allowing the concentrated solution to stand, with a resulting evaporation product containing 80 to 90 per cent of  $K_2CO_3$ . Potassium carbonate can be made by heating the bicarbonate to  $190^\circ$ , and the bicarbonate can be made by passing carbon dioxide gas through a saturated solution of the carbonate and crystallizing the product.

Potassium carbonate is used in the manufacture of hydroxide and many other potassium compounds. In 1935 the imports in terms of pure potash were 1258 tons, and the imports of bicarbonate 101 tons.

Potassium hydroxide, or caustic potash ( $KOH$ ), can be obtained by heating diluted solutions of potassium carbonate with lime at the boiling temperature but the modern method consists in the electrolysis of solutions of potassium chloride, chlorine being obtained as a by-product. Caustic potash is very soluble in water, absorbs carbon dioxide from the air, and is one of the strongest bases known. It is used in the manufacture of soft soap, shaving soap, laundry and pharmaceutical preparations, potassium chromate and other potassium compounds, oxalic acid, indigo and other dyes, organic compounds, and disinfectants, in wool scouring, and as a reagent in laboratories. In 1935, in terms of  $K_2O$ , 1370 tons of caustic potash was imported. In 1933 four American companies produced 9348 tons. In 1934 manufacturers of coal-tar products used 2400 tons in the United States.

Potassium iodide ( $KI$ ) is usually obtained by the action of potassium hydroxide on iodine or of potassium carbonate on ferrous iodide. It is used largely in medicine and photography and as a chemical reagent.

Potassium ferricyanide or red prussiate ( $K_3Fe(CN)_6$ ), is made by oxidizing a solution of potassium ferrocyanide (yellow prussiate) with chlorine. It is used chiefly in coating blueprint paper and preparing iron-blue pigments. In 1935 imports of these two salts in terms of  $K_2O$  amounted to 41 tons, and of cyanide to 7 tons.

Potassium chlorate ( $\text{KClO}_3$ ) is made by subjecting a solution of potassium chloride to the action of a direct current of electricity in an electrolytic cell. Potassium chlorate is a strong oxidizing agent and is used in manufacturing explosives, powder, fireworks and percussion caps and in toothpastes, medicines and dyes.

Potassium perchlorate ( $\text{KClO}_4$ ) contains one more oxygen atom than the chlorate and therefore possesses a greater oxidizing capacity per unit of weight in addition to being more stable. It is made by the electrolytic oxidation of the chlorate or by heating in solution sodium perchlorate with  $\text{KCl}$  and fractional crystallization. Its principal use is in railroad fuses. In 1935 imports, including chlorate, in terms of  $\text{K}_2\text{O}$  amounted to 2577 tons.

Potassium permanganate ( $\text{KMnO}_4$ ) is made by fusing manganese dioxide and potassium hydroxide and oxidizing the product in electrolytic cells to 99 per cent pure potassium permanganate. It is used largely as an oxidizer, both industrially and as an antiseptic in medicine. An important military use is as an ingredient of the filler in gas-mask canisters for the absorption of lethal gases. In 1935 the imports were 21 tons.

Potassium bitartrate [ $\text{KH}(\text{C}_4\text{H}_4\text{O}_6)$ ] is the principal constituent of wine-lees argol and of cream of tartar prepared from argol by recrystallization. Potassium bitartrate is used principally in baking powders and making other tartrates. In 1933 the American production of cream of tartar was 5789 tons, and less than one ton was imported. In 1935, in terms of  $\text{K}_2\text{O}$ , 1637 tons was imported as argol.

The alkali or soda-potash content of glass varies with the type,<sup>24</sup> to as much as 20 per cent in thin blown glass in bulbs. In several ways potash confers desirable properties on glass but its cost has limited its use in the less expensive articles. In types of glass that have a high intrinsic value, however, the cost of raw materials is not a significant factor.

The best quality crystal tableware, with high brilliance and good musical tone, is generally a straight potash lead-silica glass with 7 or 8 per cent of potash. Good chemical durability or resistance of glass to weathering is ascribed to its potash content rather than to soda. Because of the easy reduction of lead oxide in the founding process, potassium nitrate is almost universally used to maintain oxidizing conditions in this type of glass.

In X-ray lead glass, church windows and sculptured glass, practically all the alkali is potash, and to give good durability and color in optical flint glass the potash content is customarily about twice that of soda. Potash-silica glass is not appreciably affected by ultraviolet radiation, whereas soda-lime and silica glass are unstable photochemically.

In the production of colored glass decidedly different effects from colored oxides are obtained when potash replaces soda. Glass produced with potash is more colorful. When soda is put in glass for double

glazing or for insulation and other uses, a white coating often forms on the interior sides of the plates, but with 6.5 to 10 per cent of potash in the mixture the white coating does not form. For electrical insulation the part played by potash has not been adequately determined, but in a range of temperature from 100° to 400° C. there is an improvement in insulation through the use of soda and potash in the ratio of 1 to 4. For chemical wares a potash-soda glass is superior to a corresponding soda glass.

In ordinary enamelware, the potash is normally supplied by feldspar. Enamels of the jewelry type, where high brilliance and luster are required, are of the potassium-lead-silicate type with as much as 36 per cent pure potash. In the effort to replace lead as a fluxing material in glazed ware, potash may compete with lead in supplying the brilliance and luster.

#### PRICE HISTORY

The price of agricultural potash salts was increased more than tenfold during the World War, as shown by the following quotations:

In 1913 muriate was quoted at 76¢ a unit,<sup>25</sup> kainite and manure salts at 68¢. The price of potash since November 1921 has continued lower than the price in 1913, when muriate dropped to 74¢, kainite to 60¢, and manure salts to 55¢ a unit. The price of potash during the war is often said to have reached \$600 a ton, but in December 1915 it reached \$9.87 a unit, or \$987 a ton of pure potash equivalent in muriate, and \$9.05 a unit or \$905 a ton in sulphate, and did not drop below \$1 a unit for any grade from March 1915 until April 1921 and for sulphate until October 1921. During this period manure salts did not exceed \$300 a ton and kainite \$363. The price range of agricultural and industrial potash salts published by the United States Tariff Commission<sup>23</sup> are based on New York spot prices. They vary somewhat from the price quoted above but the variation is immaterial in showing the price trend.

During a part of the season of 1934-1935, potash was reported as moving freely at as low as 27¢ a unit on the Atlantic seaboard, although the lowest published price was 40¢ for muriate and 43¢ for manure salts with seasonal discounts as high as 12 per cent. In 1936 the scheduled price of 50¢ a unit for muriate with seasonal discounts is reported to be followed by all those now selling in the American market. An interesting price switch occurred in 1934 when the base price of muriate, normally 10¢ a unit above the lower grade salts, was reduced to 3¢ below. The trend of unit prices of both imports and domestic sales is shown on Fig. 2. The import price is based on seaboard delivery while the domestic price is the realization at the point of origin.

The effect of the Le Blanc soda process on potash prices is illustrated by the following price quotations: In 1810 soda ash was quoted at \$445 a ton and potash in the form of potassium carbonate at \$156; by 1850 soda

ash was \$55 and potash \$117; and by 1900 soda ash was only \$13 and potash \$90. With American sources of the material available, the manufacture of industrial potash salts, such as carbonate, should be stimulated, and the competition with foreign salts may result in a reduction from the price of \$112 a ton for crude carbonate imports in 1935, and thereby the use of potash may be increased.

Tariff on potash salts imported in America is confined to chemical and pharmaceutical grades of potassium salts. The rates imposed under the act of June 1930 are given in the Minerals Yearbook for 1930.<sup>12</sup> These range from free of duty for crude saltpeter cyanide and minus 90 per cent argols to 25¢ a pound for iodide.

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## CHAPTER XXXV

### PUMICE AND PUMICITE

By B. N. MOORE,\* MEMBER A.I.M.E.

**P**UMICE and pumicite are commercial varieties of volcanic ejecta formed from lavas of the more silicic types, such as rhyolite and dacite. The word pumice is applied to fragments of highly cellular, glassy lava. These fragments range from blocks containing several cubic feet to grains the size of sand. The completely shattered or disrupted lava is formed of small shards or bubble fragments of volcanic glass and is the volcanic ash of the volcanologist.

In 1916 Barbour<sup>2</sup> gave the name "pumicite" to the volcanic ash of Nebraska, and since then the name has been widely used in industrial circles for the finer grained commercial varieties. It is most generally applied to the fine, powdery ash of the Kansas-Nebraska region and but seldom to the coarser, sandy varieties common in the Rocky Mountain and Pacific Coast States. It has also more recently been copyrighted by the Pumicite Co. of St. Louis as a trade name. The Cudahy Co. has copyrighted the trade name "seismotite" for its product.

Chemical analyses show commercial pumicite and pumice to have the same range of composition as the more silicic lavas, and have but little, if any, bearing on their commercial use. The approximate composition of the glass can be quickly ascertained from its index of refraction as determined by use of the petrographic microscope.

The glass of pumice and pumicite is transparent and colorless and has a hardness of 5.5 to 6 and a density of about 2.5. Lump pumice is very cellular, is light gray to white, commonly with a silky sheen, and has an apparent density less than that of water. It is somewhat brittle, and lumps may be flattened or shaped by rubbing. Pumicite is very fine grained, with large fractions finer than 200 mesh and in some varieties less than 325 mesh. The material resembles a white to light gray powder but may be mixed with clay, diatomite or calcareous matter and stained. Two types of grains are distinguished, one of irregular fragments faceted by many concave fracture surfaces, the other formed of shattered filaments ribbed along their length.

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<sup>2</sup> References are at the end of the chapter.

## ORIGIN AND MODE OF OCCURRENCE

Commercial pumice and pumicite are associated with eruptions of silicic lavas. These lavas commonly contain much gas (including water vapor), in part at least as a component of a chemical system with the other rock constituents. Release of pressure and other changes tend to free the gas. The viscosity of the lava, its rate of cooling, and the amount of gas evolved all have part in determining the degree of expansion of the product and the fineness of the vesicles. If the lava is of the correct viscosity and there is not too much gas a pumice may be formed, but very large amounts of gas may cause complete disruption into pumicite.

The products of eruption are spread outward from the source in a pattern dependent on the type, direction and force of eruption, the coarseness of the particles, and the amount and direction of winds. If the products are erupted vertically the coarser materials may fall near the base of the vent and the finer materials at progressively greater distances, but if there are strong winds the finer particles may be carried great distances, particularly if projected into the upper air currents. In other deposits pumice and pumicite may be laid down in an unsorted mixture, as in eruptions of the type of Mount Pelee of the West Indies.

The extensive deposits of Colorado, Nebraska, Iowa, Kansas and Oklahoma are thought by Landes<sup>6</sup> to have originated in the Capulin Craters, of northern New Mexico. The dust was carried by air currents and deposited as a thin blanket. Subsequent wind and stream action worked it into drifts or transported it into ponds, and in some places where it was covered by loess or stream deposits and protected from later erosion it has survived to the present time.

In later Tertiary and Quaternary time widespread volcanism has occurred in the region of the Rocky Mountain and Pacific Coast States. Deposits are numerous and widely distributed in every state. Most of the deposits are local and the results of the vertical type of eruption. The extensive pumice deposits of eastern Oregon are in part the result of eruptions similar to those of Mount Pelee and in part of the normal vertical type of eruption. Because of the ease (in terms of geologic time) with which volcanic ash weathers to bentonite, the commercial deposits are generally those of Pleistocene or Recent age, which have not been subjected to much circulation of ground water. Many of the older deposits, such as those of the Gulf region and the Appalachian region, have been converted by leaching into bentonite and fuller's earth.

## DISTRIBUTION OF DEPOSITS

*United States.*—The greater part of the production of the United States has been pumicite from the deposits of Nebraska and Kansas. Less extensive deposits of both pumicite and pumice are found at many

places in the Rocky Mountain and Pacific Coast States. Pumice and pumicite have been produced from many places in California; the pumice largely from Pumice Mountain, Siskiyou County, and from the vicinity of Calipatria, Imperial County. Large amounts of a coarsely granular pumice have been produced near Laws, Mono County. This has gone largely into acoustic plasters and stuccoes. Extensive deposits of a fine white pumicite, of which 98 per cent passes a 325-mesh screen, are worked near Friant, Fresno County. The Cudahy Co. mines a volcanic ash from a bed 8 to 9 ft. thick in Last Chance Canyon, north of Saltdale, Kern County. Extensive deposits of similar ash are worked near Shoshone, Inyo County, and other deposits near Creston, San Luis Obispo County; Napa, Napa County; and Sykes, Inyo County. Commercial deposits of pumice are worked near Bouse, Yuma County, Arizona.

Deposits of pumicite related to those of Nebraska and Kansas are known in Colorado east of the Rocky Mountains. Deposits of volcanic ash are found near Creede in Mineral County, and Durango, LaPlata County, and attempts have been made to work them. Deposits of volcanic ash occur at many places in Idaho but there is no record of production. Pumicite is known to occur at many places in Montana, but there is no record of production.

Landes<sup>6</sup> has described the Kansas pumicite deposits in detail. These are most numerous in the part of the state near the 100th meridian. Farther east, according to Landes, the pumicite was removed by stream erosion, and farther west the surface was too flat to provide hollows for the accumulation of wind-drifted and stream-borne material. The deposits are from a few inches to 20 ft. thick, with great variation in individual occurrences. The overburden of soil is as much as 20 ft. thick. The pumicite is generally banded and shows marked cross-bedding. Some deposits contain considerable admixed diatomite, silt, or calcareous matter. Deposits are worked near Fowler and Meade, Meade County; Calvert, Norton County; Natoma, Osborne County; Wilson, Ellsworth County; and Satanta, Haskell County. As deposits become exhausted, the companies move the site of operations to other localities. One company is reported to have 40 deposits in reserve.

The pumicite deposits of Nebraska are very similar to those of Kansas and have been worked near Orleans, Harlan County; Ingham, Lincoln County; Edison, Furnas County; Endicott, Jefferson County; Arnold, Custer County; Scotia, Greeley County; and Atkinson, Holt County. At the present time production is recorded from deposits near Eustis, Frontier County, and Ingham, Lincoln County. The southern part of the pumicite region of Kansas and Nebraska extends into Oklahoma. Production is recorded from one deposit near Gate.

Numerous deposits of volcanic ash exist in Nevada but have not been worked. The Barnsdall Tripoli Corporation of Missouri is using a

pumice from New Mexico in the manufacture of its ground pumice abrasives. Very large deposits of pumice containing small amounts of crystals as impurity are found in Klamath and Deschutes Counties, and part of Lake County, Oregon. Deposits of pumice comparable in quality with the Italian material are found in Newberry Crater and a small area to the east of it in Deschutes County. Numerous thick beds of coarse-grained, gray volcanic ash are found in the Payette formation, near Harper, Malheur County. A commercial deposit of pumicite occurs at Winner, Tripp County, S. Dak. Deposits of pumicite have been reported at various localities in Texas. Pumicite has been found near Richfield, Sevier County, Utah, and has been reported from the vicinity of volcanic peaks in Washington, such as Mount St. Helens, and volcanic ash is common in the Ellensburg formation of the Yakima region. Volcanic ash is known to occur at many places in Wyoming, but no production has been reported.

*Canada.*—Extensive beds of pumicite occur in the vicinity of Waldeck, Saskatchewan, and Dead Man's River, Arrow Lake, Haig, and Elko, British Columbia.

*Italy.*—Pumice has been worked for many centuries on Lipari, one of the Aeolian Islands off the northeast tip of Sicily. According to Bergeat<sup>3</sup> the pumice occupies part of the northwest corner of the island and, according to Bergeat's detailed map, occupies an area of a little more than 3 square miles. It rests on older volcanic rocks and in turn is overlain by younger volcanic rocks and has a maximum thickness of about 200 meters. It is made up of dust, fragments of obsidian, partly expanded obsidian, and pumice. There is no separation of mineral grains, however, in any of these products.

*Other Countries.*<sup>5</sup>—A crudely sorted mixture of pumice and ash known as "trass" deposited in Peleean eruptions is quarried in the Rhine Valley of Germany for use largely in making building blocks. From 2000 to 4000 tons of pumicite a year is produced in New Zealand. Important deposits of pumice are reported to occur in Austria.

#### COMMERCIAL AND POLITICAL CONTROL, PRODUCTION AND CONSUMPTION

Italian pumice supplies the greater part of the European demand competing in Germany and England with the cheaper Rhine pumice, and has been preferred by users of abrasive pumice in the United States. In this country domestic pumicite and pumice fill the demand for material for cleansers, cement admixture, and acoustic stuccoes. It is probable that dissatisfaction with domestic lump pumice, aside from the relatively high freight charges from western deposits to Atlantic coast processors, has been largely due to insufficient care in grading. Domestic ground pumice from New Mexico, however, is now being sold on a basis of specified abrasive efficiency.

The production of pumice and pumicite in the United States has climbed from about 25,000 short tons in 1920 to 50,000 to 70,000 short tons in the years since 1930. Until 1916 the greater part of the pumicite was produced in Nebraska, but since then Kansas has taken the lead. Production in California has increased and since 1927 has been from about 10,000 to 14,000 short tons annually. In 1933 about 78 per cent of the total production was used in cleansers, about 15 per cent in admixtures and concrete aggregate, and about 4 per cent in acoustic plasters and stuccoes. In recent years the Italian production has risen to about 40,000 to 55,000 metric tons annually. Of this about 1 to 2 per cent is large first-quality lump (*grossa*), about 10 to 18 per cent small lump (*pezzame*) and the rest granule and ash grades (*rasaglio e lapillo*). About 60 to 70 per cent of the *pezzame* grade, or about 4000 to 6000 metric tons annually, is exported to the United States. Our imports of large lumps ranged from 74 to 203 metric tons during the years 1930 to 1933, and of ground pumice from 2433 to 3417 metric tons.

#### PROSPECTING, EXPLORATION AND MINING

Many of the deposits of the Rocky Mountain and Pacific Coast States are not covered by overburden, and prospecting has therefore not been difficult. Because of the regularity of bedding of the deposits interbedded with other sediments, large tonnages could be blocked out with little expense. The pumicite deposits of Kansas and Nebraska, however, are covered by more or less loess or alluvium and are irregular in shape and thickness. In some deposits exploration is carried on by hand auger holes put down at the intersections of a 50-ft. grid, and rather accurate estimates of quality and quantity may be made.

Methods used in mining the pumicite deposits of Nebraska and Kansas are essentially stripping operations. The overburden is removed by power shovels, and the underlying pumicite is recovered either by power shovels or by men and wagons. In places considerable care must be taken to remove concretionary masses, masses of soil that fill animal burrows in the pumicite, roots, and other material. Commonly one foot of overburden is left, particularly in intermittently operated quarries, to prevent washing and contamination of the pumicite. Deposits in the Rocky Mountain and Pacific Coast States are worked in a variety of ways, depending on their mode of occurrence. Those with little overburden are stripped and worked by open cuts. The bedded deposits in thick sedimentary formations are worked by systems of tunnels and drifts.

Operations in the Italian deposits are described in the report of the Italian minister of corporations for 1928.<sup>7</sup> Regulations restrict mining, with a few exceptions, to inhabitants, provide for rental of deposits by the community, and provide for an export tax payable to the community. Nine-tenths of the land belongs to the community. Workings are held

by workmen, either singly or collectively, and by some owners of grinding establishments, who ship the products of the independent workmen as well as their own. Workings are developed mainly by open cuts, but there are some galleries that reach depths of 50 m. These are untimbered and are sources of many accidents. The workings are dry. The loose material is mined with picks, and the lumps and dust are separated during mining. In workings some distance from the coast the dust and fines are used for fills and leveling the ground, but in those near the coast the material is air-dried and sifted to obtain a powdered pumice, which is marketed. The lumps are sorted as to size and texture, and those of poorer quality are ground in the local mills. The material is transported from the workings to the coast on the shoulders of the workmen.

#### PREPARATION FOR MARKET

The pumicite of Kansas is prepared for market by drying in rotary driers. It is screened and either packed for shipment in paper bags or shipped in paper-lined box cars. Material from Friant, Fresno County, Calif., is passed through a revolving cylinder equipped with paddles, which pulverize the lumps. This operation suffices to reduce the product to a fineness of which 98.8 per cent passes a 325-mesh screen. The granular pumice mined near Tom, Mono County, Calif., is mixed with sand. It is passed over a dry concentrating table, and the pumice thus separated is screened, and the oversize passed through a roll crusher. Material to be used with plaster is dried in rotary kilns and sacked. Most of the domestic pumice is prepared for market in the West or Midwest. The imported pumice is largely prepared on the Atlantic coast. The lumps received are crushed, sized, and packed for sale by the importer.

#### TESTS AND SPECIFICATIONS

Specifications for pumicite are not established. Most of the producers manufacture their own material into the finished products, and specifications vary with producers.

Specifications for lump pumice are also decidedly unsatisfactory, and the final test seems to be the opinion of the purchaser. It is known, however, that such material should be free from any unexpanded glass or mineral grains and should consist of small, even-walled cells. An elaborate system of grades has been established for the ground pumice, however,<sup>5</sup> and recently some producers have sold ground pumice with specified abrasive efficiencies.

#### MARKETING AND USES

The natural dust or pumicite is used largely in scouring preparations. Of the 1935 production in the United States 88 per cent went into cleansing and scouring compounds, including hand soaps; 2 per cent was used

for other abrasive purposes and the remainder found miscellaneous markets. Recently pumice has been used in increasing amounts as a cement admixture. Use of small-sized or granular pumice for acoustic stucco and plaster and for light-weight aggregate is also increasing in regions near deposits. Abrasive uses, however, face constant competition from other materials. The amount of large lump pumice imported varies and is at the most not much more than 200 tons annually. It is used largely by the lithographic trade, in automobile-body plants, and for small miscellaneous uses. Ground pumice is used largely in finishing furniture and as an abrasive in the manufacture of articles of relatively soft compositions, such as buttons. Some is used in beveling plate glass.<sup>5</sup>

It has been the experience of users of ground pumice that it is not possible to substitute the natural volcanic dust or pumicite for their uses. Ground pumice is as much as three times as efficient as pumicite of the same grade size. The difference in abrasive efficiency is due to the differences in shape of particles. Flattish striated grains are numerous in some pumicites and because of their gentle scraping action are preferred for household cleansers. The sharp, angular particles, which are more abundant in ground pumice, are more nearly the shape of grains of other abrasive materials. The uses for lump pumice have largely been supplanted by waterproof abrasive papers and cloths. Lump pumice has been used for packing in vinegar manufactories as a support for the ferment, and in chemical works as a support for various catalysts.

#### PRICES

The price of the Italian pumice is carefully controlled by exporters so as to obtain a balance between a maximum return and undue competition from other abrasives. Prevailing prices for select lump pumice f.o.b. New York are 5 to 7½¢ a pound and for ground pumice 2½¢ to 4½¢.<sup>9</sup> The value of pumicite and pumice produced in the United States increased from \$2.94 a short ton in 1920 to \$5.91 in 1930, decreased to \$3.69 in 1934, and rose to \$4.14 in 1935.<sup>9</sup>

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## CHAPTER XXXVI

### REFRACTORIES

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THE literature on refractories, although quite extensive, is so scattered through books and periodicals as to be difficult of access to the general reader, and most of the individual papers serve merely to illuminate some particular phase of the industry. Two general treatises by American authors<sup>6,13</sup> are available, also the somewhat older works by a well-known British authority,<sup>8,9</sup> but these make their appeal to the technically minded ceramic engineer. Another small group of publications deals with the use of refractories, mainly in the iron and steel industry.<sup>1,4,5,10,12</sup> Raw materials utilized by the refractories industry have likewise been covered by sundry books and articles. For the relatively nontechnical reader and economist, however, there appears to be no single review that purports to describe the industry as a whole.

Furnace linings and other products that are classed as "refractories" are designed primarily to withstand high temperatures, but this important property is by no means the only requirement. A suitable refractory material must likewise withstand slagging (chemical action), abrasion (physical wear) and spalling, as well as the various other stresses and physical phenomena induced by heat, of which softening or incipient melting is only one phase. Subject to these highly practical considerations, the definition of a refractory material, nevertheless, is based upon temperature resistance. Broadly speaking, any material that shows obvious signs of fusion after being heated slowly to 1500° C. (2732° F.) cannot be classified as a refractory material.

The characteristic properties and requirements of refractory materials are conveniently summarized in Fig. 1, prepared on the American Refractories Institute Fellowship, Mellon Institute, Pittsburgh, Pa. Most of the factors of service and properties of refractories given in this chart are interdependent and are greatly affected in degree by the functions of temperature and time. The factors in the chart are believed to cover the more common conditions encountered and naturally vary in degree, depending upon the type of service in question.

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<sup>6</sup> References are at the end of the chapter.

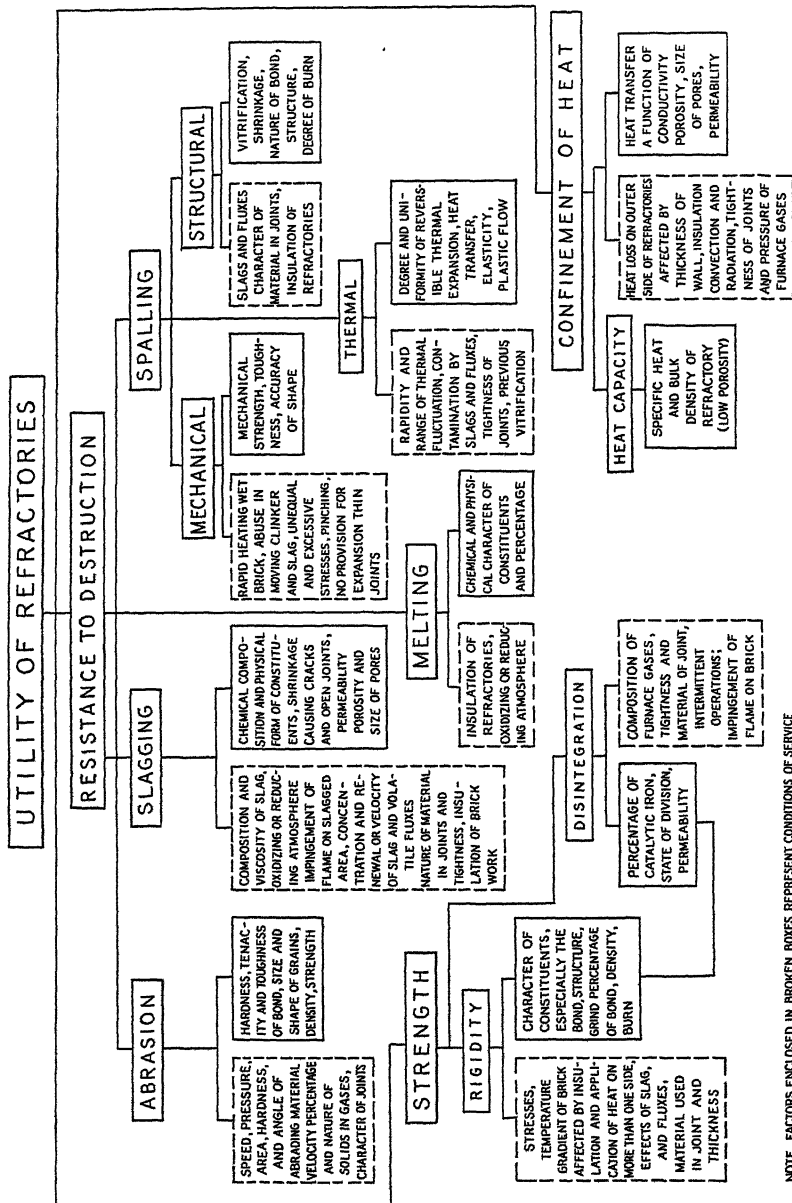


Fig. 1.—CHART FOR DETERMINING UTILITY OF REFRACTORIES UNDER DIFFERENT CONDITIONS OF SERVICE.

## CLASSIFICATION OF REFRACTORIES CONSUMERS

Various estimates have been made as to the relative importance of different industries as consumers of refractories, and the accompanying one is perhaps as good an approximation as can be made without getting average figures of actual sales over a period of years.

*Estimated Distribution of Sales of Refractories*

INDUSTRY	PERCENTAGE OF TOTAL SALES	INDUSTRY	PERCENTAGE OF TOTAL SALES
Iron and steel....	50	Glass..	5
Public utilities...	20	Oil refining ..	4
Nonferrous metals .....	6	Ceramic; miscellaneous....	3
Cement and lime ..	5	Miscellaneous ..	7

The iron and steel industry is the leading user of refractories. Refractory materials are required for the construction of blast furnaces and hot-blast stoves in the production of pig iron; of open-hearth, electric, and other furnaces used in the melting and refining of steel; soaking pits, reheating furnaces, and mill furnaces for the fabrication of the various steel products; boilers for the generation of steam; coke ovens; gas producers; and a host of other auxiliary apparatus. The public utilities and power-generating stations are the second largest consuming group. Included in this category may be considered the large central-station electric plants, individual steam-boiler plants, plants for the manufacture of city gas, railway locomotives, incinerators, sewage-disposal plants, etc. The iron and steel industry, public utilities, and other power production thus account for approximately 70 per cent of the entire consumption of refractories. The remaining 30 per cent is divided among a great variety of consumers. Prominent among them may be considered the nonferrous metal industry, which includes the smelting and refining of copper, nickel, lead, zinc, brass, and other metals or alloys. Of somewhat less importance in the consumption of refractories are the quantities used in the construction of the vertical shaft kilns and rotary kilns used for burning cement and lime. The production of glass requires refractory materials for the construction of furnaces used in the melting of glass and in the annealing and working of finished glass products. Oil refining requires the construction of stills and auxiliary apparatus, which consume important amounts of refractories products. Miscellaneous ceramic trades—such as the production of china and porcelain, sewer pipe and building brick—the manufacture of chemicals, the refining of salt and sugar, the manufacture of wood pulp and paper, all occasion the consumption of refractories. In many industries the efficiency of the processes and the capacity of the equipment, together with its useful life, are circumscribed by the ability of refractories to perform their allotted jobs. It follows that progress in the production of suitable

refractories at reasonable cost is of national concern in so far as it contributes to the betterment of many other products and reduces operating costs in our key industries.

#### TYPICAL REFRACTORIES PRODUCTS

The typical refractory product is supplied in the form of brick and preformed shapes. Standard bricks are 9 by  $4\frac{1}{2}$  by  $2\frac{1}{2}$  in., but there are numerous other so-called standard shapes, which are used in all types of construction and which, consequently, are stocked by most manufacturers. These include straight brick of different dimensions (small 9-in., split, soap and checker brick), sundry wedge, arch, skew, key, jamb, bung-arch, and neck shapes. The size tolerance on standard shapes and straight brick (American Society for Testing Materials) is 2 per cent, plus or minus, on all dimensions over 4 in.; on smaller dimensions, 3 per cent variation may be allowed. Silica standard shapes are virtually the same as firebrick, while magnesite, chrome and special or high-priced refractories are available in a more limited range of sizes and shapes. Among special shapes are various tile, segmental blocks for lining cylindrical kilns and cupolas, glass-tank blocks, coke-oven shapes, checker brick, burner tiles, boiler shapes, etc. Saggars and other forms of kiln furniture employed in the ceramic industry to protect or support ware while it is being burned account for a considerable volume of refractory shapes. Muffles of various sizes also are furnished for use in the manufacture of enamelware and in many kinds of small furnaces for heat-treating and laboratory work. Gas retorts and zinc-smelting retorts are refractory products of some importance. Crucibles are made in many shapes and sizes and from a great variety of materials; graphite-clay mixtures (6 to 50 per cent graphite) used for metal melting are the leading products in this class. Glass pots are essentially fire-clay crucibles of medium or large size used in glassmaking. Among various special refractory products may be mentioned stoppers, tubes, and such highly specialized items as laboratory ware, spark-plug cores, and other heat-resistant porcelain products.

Ordinary siliceous sand, grain magnesite, dolomite, chrome ore, and certain other materials are purchased in bulk or loose condition and rammed into place or spread evenly over the bottoms of furnaces. Various refractory cements or mortars are available for use in making joints, and plastic mixtures are employed for patching holes and general repair work and to a smaller extent in new work to form monolithic structures.

#### PRINCIPAL VARIETIES

The common refractories may be classified under the group headings Fire Clay, High Alumina, Silica, Magnesite, and Chrome. Fire-clay

refractories are the least expensive and constitute by far the largest tonnage. Silica refractories form the next larger group, their average

TABLE 1.—*Properties of Refractory Raw Materials*

Material	Approximate Formula	Melting Point, Deg. F.		Density	Hardness (Mohs)	Commercial Sources
		Pure	Commercial			
Alumina:						
Bauxite	$\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$	3722	3272-3668	2 0-2.6	1-3	Imported, Arkansas.
Diaspore.	$\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$	3722	3200-3632	3 4-3 6	6 5-7 0	Missouri.
Corundum . . . .	$\text{Al}_2\text{O}_3$	3722	3362-3686	4.0-4 1	9	Electric furnace.
Baddeleyite.. . .	$\text{ZrO}_2$	4892	2363-4505	5 5-6 0	6.5	Brazil.
Beryllia. . . . .	$\text{BeO}$	4352	4352			Rare.
Chromite. . . . .	$\text{FeO} \cdot \text{Cr}_2\text{O}_3$	3956	3200-3722	4 3-4 6	5 5	Mainly imported.
Clays:	$\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$					
Kaolin. . . . .		3245	3164-3245	2 62	2	Pennsylvania, Georgia, Alabama, Maryland
Fire clay, high-grade.			3020-2173	2 62	2	} Ohio, Pennsylvania, Missouri, etc.
Fire clay, low-grade..			2912-3002	2 62	2	
Cristobalite.....	$\text{SiO}_2$	3110	3092	2.33		See quartz.
Graphite. . . . .	C	Infus.	Infusible	2 1-2 2	1 -2	Mainly imported.
Limestone.. . .	$\text{CaO} \cdot \text{CO}_2$	4661	3803-4505	2 4-2 8	3	Common.
Dolomite.... .	$\text{CaO} \cdot \text{MgO} \cdot 2\text{CO}_2$		3497-4505	2 5-3 0	3 5-4 0	Common.
Magnesite... .	$\text{MgO} \cdot \text{CO}_2$	5072	3425-5072	3 0-3 1	3 5-4 5	Washington, California, imported.
Periclase . . . .	$\text{MgO}$	5072	3425-5072	3 7-3 9	6	Rare (also artificial).
Spinel.... .	$\text{MgO} \cdot \text{Al}_2\text{O}_3$	3875	3079-3812	3 5-4 5	7.5	Synthetic.
Quartz... .	$\text{SiO}_2$		See cristobalite	2.65	7	Pennsylvania, Ohio, Alabama, California, etc.
Diatomite . . . .	$\text{SiO}_2$		2839	0 5-1 2*	2 1*	California, Nevada, etc.
Sillimanite. ....	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$	3290	3290	3 2	6-7	British India.
Kyanite... .	$\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$	3290	3290	3 6-3 7	5-7 5	North Carolina, Virginia, California, Wyoming.
Mullite.....	$3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$	3290	3290	3 0	6-6 5	Synthetic.
Rutile.....	$\text{TiO}_2$	3344	2966	4 2-5 2	6-6 5	Virginia, Brazil, etc.
Thoria.....	$\text{ThO}_2$	5522	5522	10.		Electric furnace.
Yttria.....	$\text{Y}_2\text{O}_3$	4370	4370	5		Electric furnace.
Zircon.....	$\text{ZrO}_2 \cdot \text{SiO}_2$	4622	2452-4172	4.7	7 5	Australia, Brazil, etc.

\* Apparent; true density and hardness much higher.

price being somewhat higher than that of fire-clay products. High-alumina brick, chrome brick, and magnesite brick follow in order of increasing cost. Still more expensive are those refractories that include the synthetic materials, such as silicon carbide and fused mullite, also fused alumina, fused magnesia, and other electric-furnace products, together with the special refractories made from comparatively expensive raw materials, such as zirconia, beryllia and thoria. As the cost of production of these refractories rises, their utility becomes smaller and smaller, until certain super-refractories, like thoria, are so expensive that they can be used only in laboratory or similar small-scale work.

## FIRE-CLAY BRICK

The majority of refractories used today are classed as first-quality fire-clay brick. These usually are made of a combination of flint clays that are refractory but not plastic and highly plastic clays that are only moderately refractory. In the United States these raw materials come mainly from Pennsylvania, Kentucky and Missouri, but less extensive deposits are worked in Illinois, Ohio and Colorado. Kaolins, immense deposits of which are found in Georgia, are also employed in the manufacture of first-quality firebrick.

Fire clays vary widely in all their properties, but a classification by the American Society for Testing Materials attempts to distinguish the following classes of products for stationary boiler service: (1) high-heat-duty firebrick (above pyrometric cone equivalent 29 or 2984° F.); (2) lower-heat-duty brick in grade A (cone 28 or 2939° F.); in grade B (cone 26 or 2903° F.); and in grade C (cone 18 or 2714° F.).

First-quality fire-clay brick are used in lining blast furnaces and hot-blast stoves, checker chambers for open-hearth steel furnaces, steam-boiler settings, oil stills, and a great variety of miscellaneous furnaces. In high-temperature boiler installations and oil stills, or where severe slag conditions are encountered, brick of greater refractoriness than first-quality clay brick, and containing more alumina, are used. An entire range of high-alumina refractories has been developed to meet varying needs. The chemical composition of typical mixtures is given in Table 2.

TABLE 2.—*Typical Chemical Analyses of High-duty Fire-clay and High-alumina Brick*

Component	Composition, Per Cent				
	Firebrick, First Quality	Alumina Brick			
		50 Per Cent	60 Per Cent	70 Per Cent	80 Per Cent
Silica....	50.0-57.0	44.68	31.92	23.20	13.36
Iron oxide.....	1.5- 2.5	1.41	1.56	1.25	0.86
Alumina.....	36.0-42.0	50.37	61.04	71.25	80.21
Titania.....	1.8- 2.5	2.46	3.24	2.70	3.93
Lime.....	0.0- 0.5	0.37	0.24	0.27	0.10
Magnesia.....	0.0- 0.5	0.36	0.35	0.28	0.36
Alkalies.....	1.0- 2.5	a	a	a	a

a Not determined.

High-alumina brick are manufactured from combinations of plastic and flint clays, burleys and diaspores, a majority of which are obtained

in Missouri. The 50 per cent alumina brick is not yet in wide use but it has been substituted to some extent for first-quality clay brick in boiler furnaces, where it is more resistant to the attack of slags containing iron oxide and in the top of the checkerwork of blast-furnace stoves where fire-clay brick are attacked rapidly by the high alkali content of the entering gas.

Sixty per cent alumina brick have been substituted widely for fire-clay brick in boiler furnaces firing both oil and coal, particularly powdered coal; they are also used where refractory structures are subjected to heavy loads at high temperatures and where fire-clay brick tend to deform. The 70 per cent product is also used in lining boilers and in the gas ports of open-hearth furnaces, replacing silica brick, but by far the largest application is in lining the hot zones of rotary or vertical kilns making Portland cement or lime. Eighty per cent alumina brick are used in very limited quantities.

#### SILICA REFRACTORIES

One of the earliest types of refractory material was natural siliceous rock, rough-hewn into blocks. Sandstone blocks are still used and highly siliceous "mica schist" finds some application as furnace lining. Common sand likewise is still used to some extent in its natural state for furnace bottoms. Diatomite is another natural material, used mainly in furnaces for its heat-insulating properties and low density but also because it is refractory.

The common siliceous refractory material, however, is quartzite or "ganister." Small lump or crushed quartz and quartzite are used in the bottoms of reverberatory furnaces and as acid-converter linings in copper and Bessemer steel plants. Silica brick, however, are the principal acid refractory products employed by metallurgists. Such brick usually are made from crushed ganister bonded with a small amount (1.5 to 2 per cent) of lime. Quartzites ordinarily used in making silica brick contain about 98 per cent silica and must be otherwise suitable for the purpose. In the United States the principal deposits of ganister are the Tuscarora (Medina) quartzite of Pennsylvania, the Baraboo quartzite of the Devil's Lake region, Wisconsin, and certain quartzite beds in Alabama and Colorado. The first silica brick were made from Dinas rock in Wales, and ganister deposits near Sheffield have also contributed extensively to the refractory requirements of Great Britain; and, indeed, largely influenced the location of the iron and steel industry.

The fusion point of silica brick is virtually the same as that of the more refractory fire-clay brick, but they have other characteristics that make them more suitable for many uses. They do not soften perceptibly at temperatures approaching the melting point. Their



high thermal conductivity is also a factor contributing to their ability to retain their strength at high heats. Silica brick are used principally in the roofs of metallurgical furnaces, in coke ovens and gas retorts, and in other parts of structures where temperature changes are at a

TABLE 3.—*Typical Analyses of Silica Brick*

Component	Composition, Per Cent	
	Pennsylvania Brick (Medina Quartzite)	Western Brick (Baraboo Quartzite)
Silica .	95.67	95.50
Iron oxide . .	0.59	0.80
Alumina . .	0.92	0.98
Lime . .	2.24	2.30
Magnesia . .	0.27	0.22

minimum, where volume stability and load-bearing properties are desired, and where there is not too much corrosion from basic slags. Silica brick show a thermal expansion of about 1.0 per cent upon heating to 500° F., and this high thermal expansion is their main limitation because it causes the brick to spall or flake when suddenly heated or cooled.

### MAGNESITE

Dead-burned magnesite is the principal basic refractory material and is generally employed wherever basic slags are encountered in metallurgical furnaces at high temperature. Dead-burned magnesite is prepared from the natural magnesium carbonate or magnesite rock found in the United States in Washington and California, in several countries of Europe, and elsewhere (see chapter on Magnesite). The natural carbonate is calcined or dead-burned at a temperature of 2730° F. or higher, to expel the carbon dioxide present and convert the magnesite to a crystalline form known as periclase, which, in its pure state, has a specific gravity of approximately 3.60. The calcined product is usually red or copper brown, because of the presence of iron or other oxides. These oxides are useful in the development of the proper bond for brick manufacture and also facilitate proper sintering of the dead-burned magnesite grains when used to form the hearths of furnaces. At the same time, however, the presence of impurities is sometimes objectionable, so that there is also in use magnesite containing a higher percentage of magnesium oxide (up to 92 per cent), which must be manufactured by calcining in rotary kilns above 2900° F. or in electric furnaces.

The principal uses for dead-burned magnesite are in the manufacture of magnesite brick, also as a granular material sintered into place under

heat to form the melting hearths of basic-steel furnaces. About half of the domestic consumption of magnesite is used in the form of magnesite brick and half as dead-burned grains. Magnesite brick are used in constructing the masonry parts of basic-steel furnaces below the slag line, also many other parts of these furnaces above the slag line where the highest temperatures are encountered. Other metallurgical furnaces for smelting of copper ores, copper refining, lead smelting and refining, rotary kilns burning dolomite, Portland cement and lime, chemical furnaces, melting and treating sodium carbonate, sodium sulphate or other corrosive materials use magnesite bricks.

Magnesite brick have a higher thermal conductivity than silica brick but do not retain their strength as well when heated to a high temperature. For this reason they are not suitable for use in roofs of furnaces operating at high temperatures, except when these roofs are mechanically supported, as in the so-called "suspended-arch" construction. The field for magnesite brick promises to be expanded further by new developments in the manufacture of "unburned magnesite brick" formed from dead-burned magnesite, properly graded as to particle size, mixed with a binder, and finally consolidated under a pressure up to 10,000 lb. per sq. in. "Unburned magnesite brick" do not require kiln firing to develop the proper strength in the brick as do ordinary magnesite brick. The unburned brick develop suitable strength after drying for about 48 hr. and can be put into use immediately. The brick have strength equal to that of burned brick. They are denser and are free from warpage, from kiln marking and other defects produced in burning. The principal objection to ordinary magnesite brick has been their tendency to spall. The unburned brick are more resistant to spalling and can be used in many furnaces where burned brick have proved unsuitable. Moreover, the unburned brick cost less, which is particularly important because of the tendency in the United States to displace magnesite brick with cheaper substitutes.

TABLE 4.—*Typical Analyses of Dead-burned Grain Magnesite*

Component	Composition, Per Cent			
	Low-iron California Magnesite	Austrian Magnesite	Washington Magnesite	Quick- setting Magnesite
Ignition loss.. . . .	0.07	0.10	0.03	0.31
Silica.... .	3.96	4.50	6.90	7.35
Iron oxide.... .	0.49	3.77	4.40	6.72
Alumina..... .	0.28	1.61	2.01	2.16
Lime..... .	2.05	2.64	5.28	24.73
Magnesia ... . .	93.15	87.38	81.38	58.73

## DOLOMITE

Dolomite, a natural combination of magnesium and calcium carbonates, is much cheaper than magnesite and is used to some extent for furnace bottoms and widely for patching. The natural carbonates are calcined in rotary kilns after the manner of calcining magnesite. Other materials may be added to increase the content of iron oxide or other elements. The lime content is a disadvantage because it promotes slaking. Repeated attempts have been made to manufacture suitable dolomite brick, but today dolomite finds its principal use in dead-burned grain form or in the raw state. Although processes exist for decalcifying dolomite and converting it into nearly pure magnesia, apparently they have not yet been employed commercially. Sweetening with straight magnesite (or brucite) additions is another possibility.

Lime itself has been employed only to a limited extent as a refractory; it has a high melting point but absorbs moisture and certain gases too readily. A typical analysis of prepared dolomite is:  $\text{SiO}_2$ , 1.80 per cent;  $\text{Al}_2\text{O}_3$ , 1.10;  $\text{Fe}_2\text{O}_3$ , 5.87;  $\text{CaO}$ , 54.60;  $\text{MgO}$ , 36.40; ignition loss, 0.22.

## CHROME

Pure chromite is a mineral containing 32 per cent  $\text{FeO}$  and 68 per cent  $\text{Cr}_2\text{O}_3$ , but these oxides are often replaced in part by magnesia and alumina and the ore used for refractory purposes contains from 33 to 48 per cent  $\text{Cr}_2\text{O}_3$ , 12 to 30 per cent  $\text{Al}_2\text{O}_3$  and about 17 per cent  $\text{MgO}$ . Chrome refractories are characterized by extraordinary physical and chemical stability and for many years they were chiefly used as a neutral course between magnesite bottoms and silica-brick roofs of furnaces. Because of their high melting point and strictly neutral chemical character, their use is growing rapidly, displacing magnesite in certain fields, and also invading the field of cheaper refractories.

For a number of years, for example, magnesite brick were quoted at \$65 a short ton, as against \$45 a ton for chrome brick. The standard size ( $2\frac{1}{2}$  by  $4\frac{1}{2}$  by 9 in.) of magnesite brick weighs 10 lb. each and of chrome brick 11 lb. Chrome brick therefore cost approximately \$247.50 per 1000 compared with \$325 per 1000 for magnesite brick. In a 9-in. wall this means a saving of almost one dollar per square foot. To help meet this competition, unburned magnesite brick (using a chemical binder) were placed on the market; they were quoted at \$55 a ton, and as they are lighter than chrome brick they actually cost but little more. In 1922 the output of chrome brick was scarcely 10 per cent of the output of magnesite brick, but by 1928 the number of chrome brick produced in the United States probably at least equaled the output of magnesite brick. Despite these heavy inroads into their field of use,

the demand for magnesite refractories has continued to grow because of the increased employment of high-duty refractories instead of ordinary firebrick, silica brick, and high-alumina brick in steel, copper, and other metallurgical furnaces and in rotary cement and dolomite kilns, chemical furnaces, et cetera.

### SPINEL

Spinel is compounds of alumina with other metallic oxides in the form of aluminates, the type mineral being the magnesium aluminate ( $\text{MgO} \cdot \text{Al}_2\text{O}_3$ ) containing 28 per cent  $\text{MgO}$  and 72 per cent alumina. The melting point of the pure mineral is given as  $2135^\circ \text{C}$ . In addition to high melting point, all spinels are characterized by high specific gravity, high refractive index, extreme hardness and lack of cleavage. The natural mineral is widely distributed but rarely found in deposits of sufficient purity to make it economical to concentrate. Small seams occur in New England and New York, but the largest deposit is said to be in British India. Spinel produced synthetically in an electric furnace from oxides of magnesium and aluminum has not been used extensively because of its high cost, but in California and at the Royal Berlin Porcelain Works in Germany, brick, crucibles and various other refractory products have been produced successfully. Spinel brick also have been made by mixing equal parts of magnesite and white bauxite, calcining at  $1300^\circ \text{C}$ . or above, grading the product and forming into bricks, using a small amount of ball-clay binder, the final product corresponding to the formula of true spinel.

As a refractory, spinel has many valuable properties not possessed by either of its constituents ( $\text{MgO}$  or  $\text{Al}_2\text{O}_3$ ). It is also used in refractory cement, as an abrasive, and as a gem stone.

### SILLIMANITE, KYANITE AND MULLITE GROUP

The minerals andalusite, kyanite and sillimanite, though differing in physical properties, all have the same chemical composition; they are silicates of alumina having the formula  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ . Dumortierite is a similar mineral of (probable) composition  $8\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot \text{B}_2\text{O}_3 \cdot \text{H}_2\text{O}$ .

Sillimanite and kyanite are imported from British India and andalusite is produced commercially only in California and Nevada. Kyanite is somewhat more abundant, workable deposits being found in California, Virginia, North Carolina, Georgia, Wyoming and probably also in other states.

Mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) is a mineral of rare natural occurrence, but is formed in alumina-silica mixtures that are heated to a high temperature. It can be made synthetically by mixing the correct proportions of silica and alumina and heating above  $2012^\circ \text{F}$ . or by heating sillimanite or

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\* See also page 59.

kyanite above their decomposition temperatures. Mullite is a very desirable constituent of refractories, as it shows little or no softening below its melting point of 3290° F. The needlelike structure contributes to the high mechanical strength.

Electrically melted and cast synthetic refractories of the aluminum-silicate type, as well as mullite brick and shapes made from kyanite, are used extensively in the glass industry, and to an increasing extent in oil refineries, power plants, saggers, and sundry other situations where the disadvantage of relatively high initial cost is offset by its marked advantages; notably: (1) high melting point, (2) high thermal-dielectrical resistance, (3) low coefficient of expansion, and (4) resistance to corrosion by certain fluxing agents and furnace gases. Minerals of the sillimanite group are employed in highly refractory and tough porcelain bodies used for spark-plug cores, electrical porcelain, and laboratory, sanitary and hotel ware.

#### ZIRCONIA REFRACTORIES

Zirconia is one of the most refractory substances known and is relatively inert to molten silicate slags and other corrosive agents. Zircon, the silicate, is likewise refractory and very resistant to thermal shock but does not withstand the action of basic slags or iron oxide very well. Zirconia crucibles are now manufactured in Germany, and zirconia or zircon brick are also available. Although brittle in the unburned state, and rather costly, zirconia refractories are highly useful for certain special melting operations because of their high strength, hardness, freedom from spalling, and resistance to chemical and physical wear, mainly in precious-metal refining and in electric furnaces where exceptionally high temperatures are encountered. Zircon cement is also used as a protective coating on other refractories.

Production of zirconium minerals in the United States has been irregular and almost insignificant, except for a brief period of operations in Florida. Brazil has been the principal source of world supply of zirconia ores (zirkite, baddeleyite, etc.), but zircon is produced in increasing quantities also in British India, Australia, and elsewhere, frequently as a by-product of ilmenite mining.

#### CARBON AND GRAPHITE

Carbon is virtually infusible, and only when heated to well above 6000° F. does it exhibit any volatilization. Available forms for refractory materials are charcoal, coke and graphite. Brick made from ground coke, bonded with 20 per cent tar, dried and baked, are available, and other kinds of carbon brick have been made from time to time. Bonded amorphous carbon, pure manufactured (electric furnace) graphite, and bonded natural graphite are all on the market in various forms. Carbon

brick are used extensively in Germany for lining the hearth and bosh sections of blast furnaces producing pig iron. Carbon refractories recently have found some application in Cottrell precipitators for making acid and for furnace linings and slag floors in high-pressure boiler furnaces where pulverized coal is used.<sup>2</sup> Electric-furnace graphite is unique among refractories because of its purity, low linear expansion and nonfusibility; it retains its shape, size, structure, and physical properties under conditions of extremely high temperature. In short, carbon would be the ideal refractory if it were not for the fact that it is consumed rapidly at temperatures above bright red heat in an oxidizing atmosphere or when in contact with fused oxides of many metals.

Graphite crucibles still find extensive application in the melting of metals. They contain 25 to 75 per cent of graphite bonded with fire clay.

#### SILICON CARBIDE AND FUSED ALUMINA

Originally employed solely as abrasives, silicon carbide (Carborundum, Crystolon, Carbolite, Carbolon, Carbosilite, etc.) and fused alumina (Alundum, Adamite, Aloxite, Carboalumina, Corubin, etc.) have both come to be widely used as refractory materials. The former has no melting point and appears to be little affected by heat until above 2240° C. (4064° F.), when it dissociates into its constituent elements. It is very strong and because of its high thermal conductivity and low expansion coefficient does not spall readily. It is not attacked by acids but is attacked by alkalis, iron oxide and even molten iron and steel at high temperatures. Although rather expensive, it is used in muffles because of its high heat conductivity (from 4 to 8 times that of ordinary fire clay, depending upon the grade). Pure Carborundum brick have been used for the roofs of electric furnaces and for jamb tile, but they must be protected by an insulating material against heat and electrical shocks. It is also proposed to use silicon carbide for cooled walls in boiler furnaces as well as in recuperators and heat-regenerating systems. Silicon carbide bricks are readily interchangeable with fire-clay refractories and may be incorporated in furnace walls wherever excessive clinker formation, extreme temperatures, or high erosion conditions indicate their use. Because it is a conductor of electricity as well as a refractory, silicon carbide has also had some application as a resistor element in electrically heated furnaces, stove units, et cetera.

The enameling industries use fused alumina in the form of muffle plates and brick for lining the hot zone of continuous furnaces and those of the "box type." Fused-alumina ware in crucibles, muffles, tubes and other forms has long been employed in laboratories and small-scale high-temperature work, and despite the high cost the use of such material in electric furnaces has been seriously considered.

## REFRACTORY WARE FOR EXTRA HIGH DUTY

For certain special small-scale work demand exists for ware to withstand temperatures substantially higher than are normally encountered in commercial work, chiefly in the form of crucibles and ignition boats. Typical products have been described as follows by their manufacturers:

*Alumina* (99 per cent  $\text{Al}_2\text{O}_3$ ). Melts at  $2040^\circ\text{C}$ . ( $3740^\circ\text{F}$ .), softens above  $1900^\circ\text{C}$ . ( $3452^\circ\text{F}$ .), furnished: (1) porous in large sizes to resist temperature changes, or (2) porcelain-hard and translucent. At high temperatures more acid than neutral, not at all basic.

*Magnesia* (95 per cent  $\text{MgO}$ ). Not softening up to  $2000^\circ\text{C}$ . ( $3632^\circ\text{F}$ .), serviceable up to  $2400^\circ\text{C}$ . ( $4352^\circ\text{F}$ .), porous, and highly resistant to temperature changes. At high temperatures faintly basic or neutral; does not form carbide but vaporizes in reducing atmosphere.

*Magnesia* (99 per cent  $\text{MgO}$ ). Same refractoriness as above, but very dense and translucent, like porcelain.

*Beryllia* (99 per cent  $\text{BeO}$ ). Melts at  $2300^\circ\text{C}$ . ( $4172^\circ\text{F}$ .) and may be used above  $2000^\circ\text{C}$ . ( $3632^\circ\text{F}$ .). Porcelainlike translucent body suitable for crucibles of neutral character, replacing alumina when higher temperatures preclude use of alumina.

*Zirconia* (96 per cent  $\text{ZrO}_2$ ). Melts above  $2600^\circ\text{C}$ . ( $4712^\circ\text{F}$ .) and may be used up to  $2500^\circ\text{C}$ . ( $4532^\circ\text{F}$ .). Can be furnished in porous or denser form, according to size and shape of article. Sensitive to temperature changes but particularly resistant to chemical action of all kinds; does not vaporize appreciably in reducing or oxidizing atmosphere, but forms thin layer of carbide in contact with carbon at high temperatures.

*Thoria* (99 per cent  $\text{ThO}_2$ ). Melts at  $3000^\circ\text{C}$ . ( $5432^\circ\text{F}$ .) and may be used up to  $2500^\circ\text{C}$ . ( $4532^\circ\text{F}$ .), or above. Chemical properties similar to those of zirconia but more basic.

## MISCELLANEOUS

Titanium oxide or rutile has been formed into brick using lime as binder and is reported to have proved satisfactory as an acid refractory.

Bone ash, made by calcining bones, was formerly used extensively for cupels in assaying and refining but now is largely replaced by other materials. Fused-silica ware and porcelain are also classed as refractories, and certain varieties of glass (e.g., combustion tubing) also fall in the same category. Ceria and mixtures of the oxides of the rare earths are refractories but are far too expensive to find commercial applications.

It is not customary to consider the metals as refractories, but they are so used to a considerable extent. Ordinary cast iron, mild steel, and wrought iron are widely employed in services in which they are exposed to moderately high heats, and the new stainless and heat-resisting alloys recently developed will withstand higher temperatures. Nickel

and copper likewise find many high-temperature uses and, in a much smaller way, platinum and certain allied metals have highly important applications in the broad realm of refractory uses.

Various naturally occurring minerals and rock formations have been employed as furnace linings. Mention has been made of mica schist, sandstone and quartzite, which probably are most important, but soapstone has found applications due to the fact that it not only is refractory but also is readily sawed and otherwise formed to the desired size and shape; it has been used principally in alkali-recovery furnaces in paper mills. Talc has similar advantages, and certain varieties that exhibit no distortion when fired are employed in sundry small articles, including the so-called "lava" tips for gas burners, gas-mantle rings, etc. Pyrophyllite is likewise receiving serious consideration both in block form and in the batch (as a constituent of synthetic refractories). Olivine or dunite has been employed experimentally on a large scale to replace chromite in steel furnaces; almost unlimited supplies of this magnesium silicate and of pyrophyllite (aluminum silicate) practically free from iron are available at low cost in North Carolina. Mixed with magnesite and made into brick having a composition approaching that of forsterite, olivine has begun to be used fairly extensively.

Various mixtures of refractory materials may also be mentioned. The most important, of course, are the high-alumina fire-clay mixtures, which have already been discussed. Asbestos-clay brick are mentioned in certain textbooks as used for partitions, but inasmuch as chrysotile asbestos is altered and embrittled at temperatures below the firing temperature of most firebrick, such mixtures are of doubtful value. Insulating brick and cements (containing diatomite, vermiculite or mineral wool, or ordinary firebrick compositions rendered porous) are of rapidly increasing importance but most of them cannot resist very high temperatures and so must be protected from the fuel heat of the furnace by a layer or course of truly refractory material.

#### RAW MATERIALS

According to the raw materials required, the refractory industry is roughly separable into the manufacture of fire-clay and silica refractories as a major group and the production of other refractories as a minor group. In general, the former group comprises plants producing relatively cheap tonnage products, while the latter comprises the highly diversified industry that caters to the demand for high-duty refractories to meet more exacting requirements, which therefore cost more. Compared with the ordinary run of heavy clay products, however, even the cheapest refractory material is relatively high in price; and ordinary firebrick grade more or less insensibly into high-duty firebrick, as better and more expensive materials or mixtures of materials are employed.



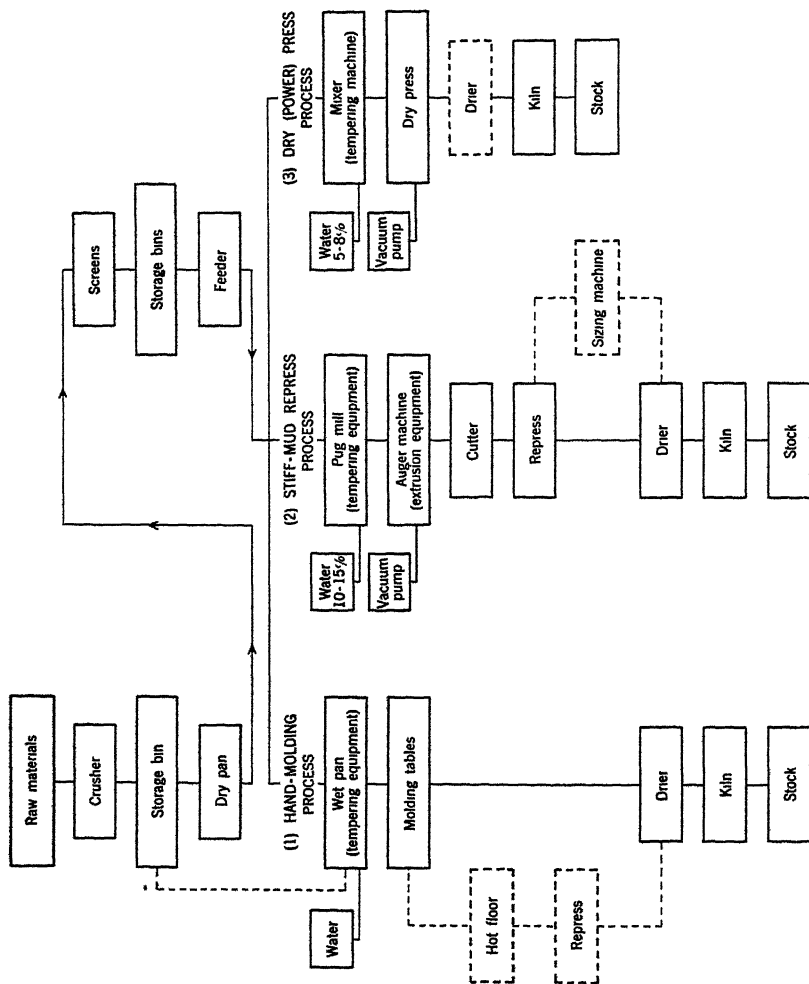


FIG. 2.—THREE METHODS OF MANUFACTURING FIRE-CLAY BRICK.

There is an economic point in attempting to draw a distinction, however, to the extent that a cheap raw material like ordinary fire clay must find its market fairly close to the point of origin, whereas magnesite and chrome are carried halfway around the world from the mines to the brickworks.

## METHODS OF MANUFACTURE

### FIRE-CLAY BRICK

The three general methods used in manufacturing fire-clay brick are hand molding, stiff-mud repressing, and dry pressing. Hand molding is used mainly in the manufacture of shapes that are very large or have very irregular contour. The bulk of fire-clay brick is manufactured by the other two processes, and the choice between them depends upon the nature of the raw materials and the properties desired in the finished brick. The various manufacturing steps involved in the three processes are shown in the flowsheet (Fig. 2).

The three principal raw materials are flint clay, bond clay, and grog or calcine. The flint clay makes up the bulk of the brick. It is very hard and refractory. Some flint clays can be processed without the addition of bond clay; others cannot. Bond clay, a plastic material somewhat less refractory than flint clay, is used mainly to impart bond or strength to the brick to facilitate processing and handling in the green state. Where flint clays are extremely low in plasticity the use of bond clay also helps in the burning operation. Grog or calcine is used mainly to control shrinkage in burning. Grog may consist of brick that have been ground to the required screen analysis or may be a clay that is calcined in the lump form as it comes from the mines and then crushed or ground.

As shown in the flowsheet (Fig. 2), the general method of preparing the clays for any of the three processes of manufacture is about the same up to the molding operation. Occasionally, where the clays being ground and prepared are very uniform in hardness, the mix for hand molding may be ground directly in the wet pan or tempering machine. Some hand-molded shapes are allowed to dry somewhat and are repressed to improve workmanship; others are merely touched up at the corners and set directly from the molding tables into the drier.

In the stiff-mud repress process some manufacturers put the green brick through a sizing machine to improve the workmanship. In the manufacture of dry-press brick it is the general practice to set the brick right from the press into the kiln, although sometimes a drier is used. Dry-pressed brick are somewhat cheaper to manufacture than stiff mud. The brick are formed under relatively high pressures, ranging from 1000 to 4000 lb. per sq. in., and have virtually no drying shrinkage.

Fire-clay brick manufactured by the stiff-mud repress method are usually harder, tougher, and denser than dry-press brick. They are used where resistance to erosion by molten slag or resistance to abrasion are the outstanding requirements.

Brick made by any of the three processes are fired both in tunnel and periodic kilns. Where large, steady production is available the tunnel kilns are economical. Fire-clay brick are burned at temperatures anywhere from 2300° to 2550° F., again depending upon the nature of the raw materials and the properties desired in the finished brick.

### SILICA BRICK

Three general processes are used for the manufacture of silica brick—hand molding, machine molding, and dry pressing. The various steps involved in each method of manufacture are shown in Fig. 3.

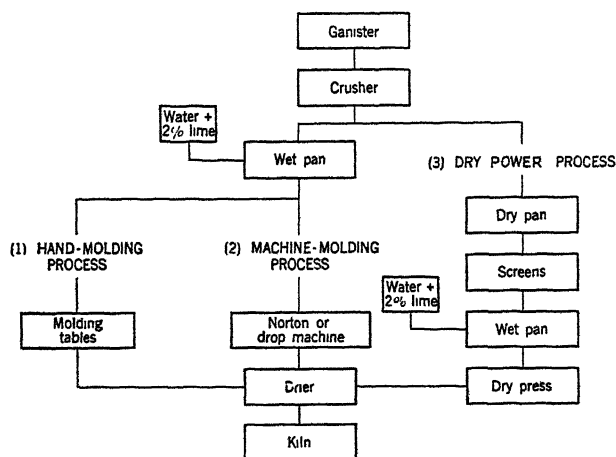


FIG. 3.—THREE METHODS OF MANUFACTURING SILICA BRICK.

Ganister, the essential raw material, is a massive crystalline quartzite. It is broken down in crushers of the jaw or gyratory type, and in the hand and machine molding process is ground to the desired screen size in a wet pan, during which operation approximately 2 per cent of lime is added. In hand molding the shapes are prepared by pounding the mix into molds with heavy mauls. In machine molding the mix from the wet pan is sent to an overhead bin and dropped about 15 ft. into the molds on a molding table below. In the dry-press process, which is comparatively new, the crushed ganister is ground dry in a dry pan or other grinding mill and screened to the desired screen analysis. Pressing is done on a machine similar to that used for the manufacture of fire-clay brick.

Silica brick are very difficult to fire satisfactorily and must be handled carefully because of some of the sudden expansion changes during burning.

Firing usually is done at about 2700° to 2750° F. in periodic kilns. One tunnel or continuous kiln, however, is now in operation in this country. Machine molding is used mainly for small shapes of regular contour. Hand molding is used for the larger shapes, and the bulk of the silica-brick production is made by this method of manufacture. The dry press can make larger shapes than the machine and also improves workmanship. The properties of the finished brick will vary somewhat, according to the nature of the raw material.

### MAGNESITE BRICK

Two general types of magnesite brick are available, burned and unburned. The latter has assumed a position of major importance in magnesite refractories within the last 5 or 6 years. The flowsheet in Fig. 4 shows in general the steps involved in the manufacture of burned brick and the two types of unburned brick.

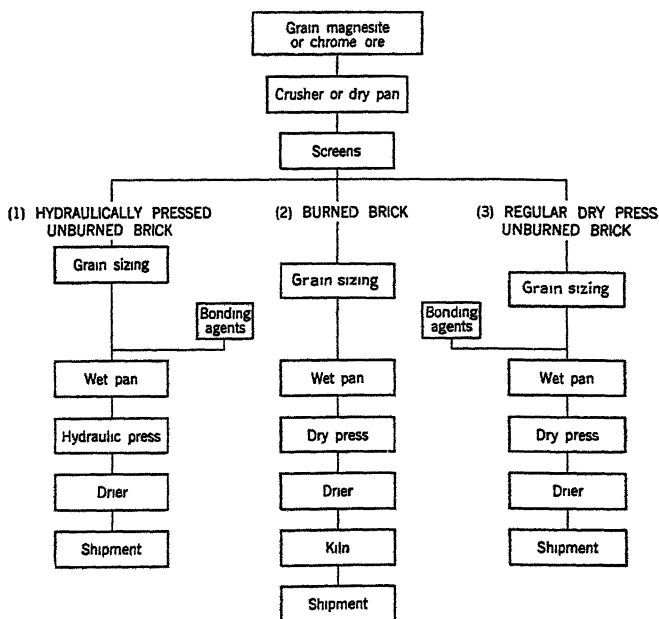


FIG. 4.—MANUFACTURING METHOD FOR BURNED MAGNESITE AND CHROME BRICK AND TWO METHODS FOR UNBURNED BRICK.

The principal raw material is dead-burned grain magnesite. This is ground in a dry pan or other suitable mill and screened carefully. For hydraulically pressed unburned brick, where extremely high pressure is used in pressing, the ground material must be closely sized and proportioned. The mix is prepared for pressing in conventional wet pans. The brick, except those made on the hydraulic press, are formed in a

conventional dry press similar to that used in the manufacture of fire-clay and dry-press silica brick. After pressing, the brick are dried, and the unburned brick are put into stock immediately. The brick to be fired are set in periodic kilns and burned to about 2700° F. Firing requires a great deal of care, and the brick must be set so that they support only their own weight. Individual brick are surrounded by silica brick, so that half the space in the kiln under fire is filled with silica brick that have already been burned and are of no value.

The hydraulically pressed unburned magnesite brick, owing to careful grain sizing and the high forming pressures used, are denser and heavier than the others and shrink less in service. Their strength is satisfactory despite the fact that kiln firing has been omitted. The cost of the unburned brick is considerably less than that of burned brick. Unburned brick are frequently laid into furnace walls with iron plates or envelopes about  $\frac{5}{64}$  in. thick surrounding each brick. These plates increase the resistance of the brick to spalling and thereby prolong their life. An older form of obtaining a similar result consisted in ramming ground magnesite into cylindrical or rectangular metallic containers, which then were built into furnace walls as desired.

#### CHROME BRICK

The bulk of the chrome brick production is manufactured in much the same way as magnesite brick. Two general types of chrome brick are available, burned and unburned. Some chrome brick are being fired in tunnel kilns and one manufacturer has developed a process in which the chrome ore, before grinding or screening, is calcined in a rotary kiln. This particular brick is fired in a tunnel kiln at temperatures considerably higher than those normally used. It contains about 20 per cent of dead-burned grain magnesite, which greatly improves the physical properties of the chrome-magnesite brick as compared to ordinary chrome brick. Similar mixtures of chrome and magnesite are available in hydraulically pressed unburned brick and their physical properties are also better than ordinary chrome brick, particularly with respect to their resistance to spalling.

#### HIGH-ALUMINA BRICK

Highly aluminous refractories are manufactured in much the same general way as fire-clay brick, except that burley and diaspore clays, obtained mainly in the State of Missouri, are substituted for flint clay, and the formed brick are burned at higher temperatures, generally about 2700° F. For special purposes other highly aluminous refractories are made by using kyanite or sillimanite instead of burley or diaspore.

## MISCELLANEOUS REFRACTORIES

*Zircon or zirconia brick* usually are molded in a dry press from crushed ore, fines of the same material proving the best binding agent.

*Silicon carbide bricks* are made generally from properly sized grains bonded with fire clay, although it is possible to produce a recrystallized product composed entirely of silicon carbide from previously made grains cemented by a temporary organic binder and reheated in the furnace.

*Cast blocks* are made by at least one domestic manufacturer, who actually melts alumina-silica mixtures in an electric furnace and casts the product in molds. Suitable arrangements must be made for slow cooling and annealing to develop the desired structure and mechanical properties.

## HISTORY OF THE INDUSTRY

Ancient Egyptian and Assyrian records indicate that the manufacture of clay brick was one of the earliest established industries, and the use of refractories of a sort has been a necessary accompaniment of all high-temperature processes since the beginning of man's use of fire. However, the early metallurgists built their own furnaces from available materials, and the manufacture of refractories did not emerge as a separate industry of importance until comparatively recently. The zinc-smelting industry still produces some of its own refractories, notably retorts and condensers.

In colonial days the iron furnaces and primitive forges were constructed of stone, and firebrick did not come into general use, in the United States at least, until well along in the nineteenth century. Sandstone was used even for the walls of kilns burning refractories.<sup>11</sup> Norton (ref. 6, p. 12) infers that the first clay refractories used in this country were glass pots, and German pot clays were imported and probably used more or less extensively in the various glass plants built before the American Revolution, many of which were operated by Germans. The manufacture of glass pots as an industry separate from the glass plants was begun in Pittsburgh<sup>7</sup> in 1860 by Thomas Coffin. The Pittsburgh Clay Pot Co. was organized in 1879, and subsequently many other concerns opened for business in Pennsylvania and Ohio.

The production of firebrick in the United States probably dates back to 1812, by a factory said to have existed in New Jersey, but firebrick may have been manufactured in Boston or Baltimore some time before that. In 1837 the Mt. Savage fire clay was discovered, but firebrick were being made in Baltimore before that date, and Ries and Leighton<sup>7</sup> state that records show a shipment of firebrick from Florida to New Orleans in 1827. The Ohio industry was established at East Liverpool in 1841, but by that time the New Jersey and Pennsylvania industries had been in steady operation for some years. Several firebrick companies were in business in Missouri in 1855, and shortly after the Civil War (in

1866) firebrick were being made at Golden, Colo., which remained the western outpost of the industry for several decades, the California and Washington plants being of relatively recent establishment.

According to Norton (ref. 6, pp. 19-20), probably the first silica brick made in the United States were manufactured at Akron, Ohio, in 1866; later they were made at Niles, Ohio, in 1872, and at Perth Amboy, N. J., in 1875, although the modern lime-bonded silica brick is a more recent development. Steel manufacturers began to use chrome brick about 1896. Magnesite was not successfully used in the United States until 1888, although it was in regular use in Europe about 1880. High-alumina refractories are of recent development, and Missouri diaspore was not recognized as a source of material for super-refractories until 1917. Sillimanite, kyanite and mullite refractories were all developed after the close of the World War.

#### WORLD PRODUCTION

The production of firebrick and other refractories is well established in Germany, Belgium, Great Britain, France and Italy, as well as in the United States, and various other industrial countries have refractory-producing industries of at least local importance. There is a natural tendency to develop local industries wherever suitable raw materials are available and a market exists. Magnesite brick, for example, are made in substantial quantities in Austria and Czechoslovakia for home consumption and export; and in Chile, according to a Department of Commerce report,<sup>3</sup> firebrick are produced by four firms, whereas before 1932 the smelters of that country depended wholly upon foreign sources. In at least two countries—Belgium and Poland—the existence of suitable clay deposits accompanied by fuel supplies encouraged the establishment of important zinc-smelting industries that otherwise might have been located elsewhere. Even in the British Isles there is reason to believe that the many occurrences of good refractory materials have been a favorable factor in the development of local iron and steelworks and other industries that utilize refractories in high-temperature processes. The growth of a flourishing refractories-manufacturing industry in Germany was due in part to the possession of a few beds of superior clays but perhaps to an even greater extent to thorough development and organization.

#### DOMESTIC INDUSTRY

##### PRODUCTION

Complete statistics showing the total output of refractories in the United States are not available, and any aggregate figures that may be compiled are subject to discussion as to the character and variety of items included. Chemical porcelain and chemical stoneware, for example, may

TABLE 5.—*Production of Firebrick and Allied Fire-clay Products, by Kinds and States, 1929 and 1932<sup>a</sup>*

Kind and State	1929			1932		
	Number of Plants	Quantity, Thousands	Value, Thousands of Dollars	Number of Plants	Quantity, Thousands	Value, Thousands of Dollars
Brick, block or firebox tile (9-in. equivalent):						
Total.....	198	938,218	\$36,169	178	217,412	\$7,611
California.....	16	26,714	1,830	18	8,283	372
Colorado.....	6	16,083	595	7	6,787	239
Illinois.....	3	25,216	796	4	3,823	88
Indiana.....	4	5,867	136	2	b	b
Kentucky.....	11	101,818	4,474	9	16,524	603
Missouri.....	13	158,505	5,968	11	47,902	1,588
New Jersey.....	10	17,450	928	7	3,809	175
Ohio.....	25	152,175	3,966	23	28,564	660
Pennsylvania.....	59	352,421	14,897	56	68,831	2,793
Texas.....	7	6,799	223	5	1,031	18
Washington.....	5	6,715	254	3	b	b
Other states <sup>c</sup> .....	39	73,455	2,601	33	b	b
High-alumina (over 40 per cent) brick: total..	17	25,911	2,115	12	4,864	464
Missouri.....	6	6,716	542	5	2,506	251
Ohio.....	4	8,175	469	3	732	38
Other states <sup>d</sup> .....	7	9,020	1,104	4	1,626	175
Special shapes: total.....	53	Tons 182,280	4,092	73	Tons 85,998	1,848
California.....	7	6,961	196	12	4,005	90
Missouri.....	7	76,418	1,532	8	29,252	520
New Jersey.....	4	5,551	263	5	8,551	223
Ohio.....	6	12,333	298	10	12,320	270
Pennsylvania.....	15	50,416	1,085	13	9,357	268
Other states <sup>e</sup> .....	14	30,601	719	25	22,513	477
Glasshouse tank blocks, pots, etc.: total..	13	35,912	2,276	16	15,869	908
Indiana.....	2	b	b	3	2,259	137
Pennsylvania.....	5	8,124	545	4	b	b
Other states <sup>f</sup> .....	6	b	b	9	b	b
Refractory cement (clay): total.....	48	57,532	1,693	51	21,766	866
California.....	6	5,306	88	4	b	b
Illinois.....	2	b	b	3	61	2
New Jersey.....	4	b	b	8	5,562	233
New York.....	6	b	b	4	406	27
Ohio.....	4	2,292	84	7	982	33
Pennsylvania.....	12	b	b	11	5,466	163
Other states <sup>g</sup> .....	14	b	b	14	b	b
Clay, raw or prepared: total.....	229	464,215	2,300	149	134,815	686
California.....	16	12,860	104	15	3,137	38
Colorado.....	5	4,662	14	5	1,035	5
Illinois.....	3	28,539	64	5	b	b
Indiana.....	6	12,561	48	2	b	b
Iowa.....	5	2,289	24	5	1,150	9
Kentucky.....	9	33,714	113	8	4,175	26
Massachusetts.....	10	3,053	22	3	410	6



TABLE 5.—(Continued)

Kind and State	1929			1932		
	Number of Plants	Quantity, Thousands	Value, Thousands of Dollars	Number of Plants	Quantity, Thousands	Value, Thousands of Dollars
Missouri.	11	77,073	542	12	36,867	212
New Jersey.	13	27,749	217	5	2,090	19
Ohio....	33	114,762	373	20	34,695	93
Pennsylvania.	51	101,588	504	30	18,627	138
Other states <sup>a</sup> .	62	45,365	178	39	b	b
Other clay products: total.	71	b	3,503	41	b	694
Illinois.	8	b	950	6	b	77
Ohio..	18	b	1,467	9	b	366
Other states <sup>a</sup> .	45	b	1,086	26	b	251

<sup>a</sup> Compiled from reports of the U. S. Bureau of the Census

<sup>b</sup> Data not available.

<sup>c</sup> In 1929: Alabama, 2 plants; Arkansas, 1; Connecticut, 1; Georgia, 3; Idaho, 2; Maryland, 5; Massachusetts, 2; Michigan, 2; Minnesota, 1; Montana, 2; New Mexico, 1; New York, 4; North Dakota, 2; Oregon, 1; South Carolina, 1; Tennessee, 2; Utah, 1; Virginia, 1; West Virginia, 5. In 1932: Alabama, 3; Maryland, 4; New York, 3; New Mexico, 2; North Dakota, 1; South Carolina, 3; Tennessee, 1; West Virginia, 4; other states same as in 1929; except no output in Michigan, Minnesota, Oregon, or Utah.

<sup>d</sup> In 1929: California, 2 plants; Colorado, 1; Georgia, 1; Illinois, 2; Pennsylvania, 1. In 1932: California, 1; Illinois, 1; Pennsylvania, 2.

<sup>e</sup> In 1929: Arkansas, 1 plant; Colorado, 2; Georgia, 2; Idaho, 1; Kentucky, 2; Maryland, 1; Massachusetts, 2; New York, 1; Washington, 2. In 1932: Colorado, 4; Georgia, 1; Idaho, 1; Illinois, 2; Indiana, 1; Kentucky, 4; Maryland, 1; Massachusetts, 2; Michigan, 1; New York, 2; South Carolina, 1; Texas, 1; Utah, 1; Washington, 3.

<sup>f</sup> In 1929: California, 2 plants; Missouri, 1; New Jersey, 1; Ohio, 2. In 1932: California, 3; Missouri, 3; New Jersey, 1; Ohio, 2.

<sup>g</sup> In 1929: Alabama, 1 plant; Indiana, 1; Massachusetts, 2; Michigan, 1; Missouri, 7; South Carolina, 1; Vermont, 1. In 1932: Alabama, 1; Colorado, 1; Georgia, 1; Massachusetts, 1; Michigan, 1; Missouri, 4; Montana, 2; Nebraska, 1; Washington, 2.

<sup>h</sup> In 1929: Alabama, 3 plants; Arkansas, 1; Connecticut, 2; Georgia, 1; Idaho, 1; Maine, 2; Maryland, 5; Michigan, 1; Minnesota, 3; Montana, 3; Nebraska, 3; New Mexico, 2; New York, 6; North Dakota, 2; Oklahoma, 1; Oregon, 2; South Carolina, 1; Tennessee, 2; Texas, 7; Utah, 2; Virginia, 1; Washington, 3; West Virginia, 6; Wisconsin, 1; Wyoming, 1. In 1932: Alabama, 3; Connecticut, 1; Georgia, 2; Idaho, 2; Maine, 3; Maryland, 3; Michigan, 2; Nebraska, 2; New Mexico, 1; New York, 4; Oklahoma, 1; Oregon, 1; South Carolina, 2; Tennessee, 1; Texas, 5; Utah, 1; Washington, 3; West Virginia, 1; Wyoming, 1.

<sup>i</sup> In 1929: Alabama, 1 plant; California, 13; Colorado, 1; Georgia, 1; Indiana, 3; Iowa, 3; Kentucky, 1; Michigan, 1; Missouri, 3; New Jersey, 3; New York, 1; North Dakota, 1; Oklahoma, 1; Oregon, 1; Pennsylvania, 6; Tennessee, 1; Texas, 2; Utah, 1; Virginia, 1. In 1932: Alabama, 3; California, 5; Colorado, 2; Iowa, 1; Kansas, 1; Maryland, 1; Minnesota, 1; Missouri, 1; New Jersey, 1; New York, 1; Pennsylvania, 6; Texas, 1; Utah, 1; Wyoming, 1.

be classed as refractories; and there are numerous articles, such as saggars or zinc retorts, that ordinarily are made by the consumer for his own use. Any statements as to the expansion of total domestic production therefore, are subject to modification, owing to changes in the variety of items classed as refractories for which statistics are available. Moreover, the apparent expansion is to a small but appreciable extent due to increased coverage in statistical canvasses.

From available data, however, it appears that under predepression conditions the aggregate value of the products of the domestic refractories industries was of the order of \$90,000,000 to \$100,000,000, or roughly

TABLE 6.—*Production of Fire Clay in the United States, 1900–1935<sup>a</sup>*

Year	Short Tons	Value	Year	Short Tons	Value
1900	845,559	\$947,993	1918 . . . . .	2,305,033	\$5,664,064
1901. . . . .	929,749	1,514,508	1919 . . . . .	1,755,331	4,628,605
1902. . . . .	926,890	891,185	1920. . . . .	2,261,915	7,088,049
1903. . . . .	1,183,926	1,474,260	1921. . . . .	1,195,861	3,560,373
1904. . . . .	1,068,598	1,306,053	1922. . . . .	1,679,220	4,633,486
1905 . . . . .	1,229,647	1,529,468	1923. . . . .	2,298,163	6,565,899
1906 . . . . .	1,380,472	1,878,011	1924 . . . . .	2,443,710	6,737,063
1907. . . . .	1,474,462	2,054,698	1925 . . . . .	2,566,934	7,312,349
1908. . . . .	1,101,579	1,486,139	1926 . . . . .	2,819,346	8,119,323
1909. . . . .	1,463,919	2,082,193	1927 . . . . .	2,699,761	7,719,725
1910. . . . .	1,638,931	2,157,720	1928. . . . .	2,785,158	7,480,609
1911. . . . .	1,526,921	2,112,827	1929. . . . .	3,178,805	8,107,586
1912 . . . . .	1,695,337	2,363,357	1930 . . . . .	2,547,162	6,070,663
1913. . . . .	1,820,379	2,592,591	1931 . . . . .	1,473,161	3,741,038
1914 . . . . .	1,409,467	2,147,277	1932. . . . .	725,993	2,057,060
1915 . . . . .	1,570,481	2,361,482	1933 . . . . .	1,133,693	3,141,545
1916. . . . .	2,057,814	3,708,009	1934. . . . .	1,288,909	3,733,033
1917. . . . .	2,347,972	5,625,095	1935 . . . . .	1,903,027	5,040,808

<sup>a</sup> Sales by producers as reported in Mineral Resources of the United States and the Minerals Yearbook (annual volumes); does not include kaolin, ball clay, bentonite, and certain other clays used in manufacture of firebrick, fire-clay mortar, or other refractory uses.

TABLE 7.—*Sales of Raw Clay for Specified Refractory Purposes<sup>a</sup>*

SHORT TONS, COMPARED WITH TRADE INDEXES

Use	1923–25 Average	1926–28 Average	1929	1930	1931	1932	1933	1934	1935
Gas retorts. . . . .	7,757	2,157			979	131	1,487		
Zinc retorts, etc. . . . .	49,260	65,299	43,034	13,174	5,961	6,298	14,427	20,399	9,988
Crucibles. . . . .	12,800	3,309	2,430	936	938	252	238	637	329
Glass pots. . . . .	31,826	25,194	49,258	40,066	27,253	20,227	2,579	598	1,585
Other glass refractories	19,665	1,346	10,221	12,217	1,068	794	4,381	10,152	929
Firebrick and block. . . . .	739,253	971,481	1,232,560	1,043,769	601,588	297,839	571,118	684,550	1,043,311
Fire-clay mortar. . . . .	392,385	444,304	406,905	443,973	233,562	128,731	171,285	172,455	239,250
High alumina brick. . . . .	891	4,479	3,811	4,656	11,050	2,700	6,547	9,416	2,328
Foundries, steelworks . . . . .	517,041	508,205	620,210	362,305	219,002	118,558	258,503	296,398	345,222
Total . . . . .	1,770,978	2,028,774	2,418,229	1,931,096	1,101,401	573,530	1,030,565	1,194,655	1,643,942

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Raw clay sales <sup>b</sup> . . . . .	100	114	137	109	63	32	58	87	92
Fire clay <sup>c</sup> . . . . .	100	114	130	105	60	30	46	52	77
Iron and steel production. . . . .	100	112	130	94	59	31	54	80	79
Industrial activity. . . . .	100	108	119	96	81	64	76	79	90

<sup>a</sup> As reported by producers to the Bureau of Mines.

<sup>b</sup> Sales of all kinds of clay to refractories industries, as listed above.

<sup>c</sup> Total sales of fire clay only to all industries.

equal to the normal output of pottery products in the United States. The number of employees has been estimated at around 20,000. Plant capacity is vastly in excess of domestic needs, even in active years. Attention should be drawn, moreover, to the close correlation between the refractories industries and iron and steel production. Shipments of fire clay as reported by the Bureau of Mines constitute an even better barometer of the state of the refractories industries, as may be seen in Table 7.

### GEOGRAPHICAL DISTRIBUTION OF PRODUCTION

Pennsylvania is by far the leading source of domestic refractory products, furnishing in recent years substantially more than one-third of the clay firebrick and almost three-fourths of all the silica brick produced in the United States, as well as much of the magnesite brick, chrome brick, and other special refractories. Of a total of 394 establishments listed by the American Refractories Institute, 106 were in Pennsylvania. Missouri, Ohio and Kentucky normally follow Pennsylvania in about the order named. The industry is represented in at least 33 states, but the four leading states account for roughly 80 per cent of the total

TABLE 8.—*Production of Specified Refractory Products<sup>a</sup>*

Kind	1925-1929, Average			1932		
	Plants	Quantity	Value, Thousands of Dollars	Plants	Quantity	Value, Thousands of Dollars
Clay products, thousands:						
Brick, block, or firebox tile (9-in. equivalent). . .	219	931,481	\$36,947	178	217,412	\$7,611
High-alumina brick (over 40 per cent $Al_2O_3$ ) . . . . .	14	12,637	1,211	12	4,864	464
Special fireclay shapes, tons.	43 <sup>b</sup>	115,870 <sup>b</sup>	2,756 <sup>b</sup>	73	85,998	1,849
Glasshouse tank blocks, pots, tons	13 <sup>c</sup>	37,534 <sup>c</sup>	2,193 <sup>c</sup>	16	15,869	908
Refractory cement (clay), tons .	43 <sup>b</sup>	46,322 <sup>b</sup>	1,299 <sup>b</sup>	51	21,766	866
Clay sold raw or prepared, tons	237	518,351	2,738	149	134,815	686
Other clay products, tons	86 <sup>d</sup>		5,004 <sup>d</sup>	41		694
Silica brick, thousands . . . . .	28	256,818	13,001	25	40,111	1,762
Magnesite and chrome brick, thousands . . . . .	5	14,864	4,433	3	3,167	845
Graphite crucibles, <sup>f</sup> tons . . . . .	11		2,336	9	2,127	639
Refractory cement (nonclay), tons .	20 <sup>b</sup>	64,255 <sup>b</sup>	1,337 <sup>b</sup>	24	16,693	444
Others, including alumina and silicon carbide refractories, tons. . . .	19		5,257	29		2,414

<sup>a</sup> Compiled from U. S. Bureau of the Census reports. <sup>d</sup> Includes some duplication.

<sup>b</sup> Four-year average.

<sup>c</sup> Data not available.

<sup>e</sup> Three-year average.

<sup>f</sup> Average 1925, 1927, and 1929.

quantity of firebrick and an even larger percentage of the total value of all refractory products. The reason for the concentration of the industry in a relatively small number of important centers is indicated in part by statistics of fire-clay production, which show that recently more than two-thirds of the tonnage of this important raw material has been mined in the four states mentioned. Nearness to markets in the leading iron and steel districts and transportation facilities are other factors.

TABLE 9.—*Production of Refractory Brick in the United States, 1911–1936*<sup>a</sup>

Year	Clay Firebrick		High-alumina Brick (over 40 per cent)		Silica Brick		Magnesite and Chrome Brick	
	Thousands	Value, Thousands of Dollars	Thousands	Value, Thousands of Dollars	Thousands	Value, Thousands of Dollars	Thousands	Value, Thousands of Dollars
1911	705,021	\$13,554	b	b	104,483	\$2,521	b	b
1912	778,103	14,954	b	b	135,578	2,923	b	b
1913	843,053	16,811	b	b	174,246	3,816	b	b
1914	687,091	13,476	b	b	129,693	2,952	b	b
1915	833,739	15,800	b	b	130,244	3,040	b	b
1916	1,166,340	24,437	b	b	232,673	6,369	b	b
1917	1,304,286	42,502	b	b	327,030	15,511	b	b
1918	1,222,352	51,648	b	b	336,562	19,988	b	b
1919	963,439	38,016	b	b	211,420	10,915	b	b
1920	1,114,809	53,416	b	b	250,532	15,077	b	b
1921	621,222	24,833	b	b	104,990	5,221	b	b
1922	836,724	31,357	b	b	186,547	7,533	b	b
1923	1,134,223	46,677	b	b	258,388	12,855	12,900 <sup>c</sup>	\$4,060
1924	955,399	40,621	b	b	204,338	10,084	12,800 <sup>c</sup>	3,833
1925	955,079	41,164	4,966	\$436	235,877	11,280	12,732	3,752
1926	1,016,879	40,992	9,095	979	266,409	13,614	16,130	4,763
1927	877,748	35,474	11,351	1,187	245,881	12,757	13,941	3,874
1928	868,755	34,874	13,860	1,336	241,523	12,188	14,116	4,144
1929	938,218	36,169	23,911	2,115	294,402	15,165	17,399	5,631
1930	758,934	29,737	11,833	1,592	212,640	11,523	13,432	3,878
1931	416,041	15,686	7,020	734	103,557	5,132	8,565	2,234
1932	217,412	7,611	4,864	464	40,111	1,762	3,167	845
1933	372,523	13,116	9,553	718	110,889	4,655	9,027	2,580
1934	390,214	15,485	15,719	1,024	103,534	5,705	10,640	3,092
1935	481,679	19,496	13,220	1,071	149,621	8,180	12,112	3,425
1936	615,498	26,580	11,151	1,122	229,325	12,453	20,403	5,767

<sup>a</sup> Compiled from reports of the U. S. Geological Survey and U. S. Bureau of the Census.

<sup>b</sup> Not separately reported. <sup>c</sup> Estimated from tonnage.

#### ORGANIZATION OF DOMESTIC INDUSTRY

Clay firebrick were manufactured in 1929 in 198 establishments, and the American Refractories Institute listed (in 1930) 147 companies operating 222 refractories plants in 27 states. A considerable portion of the

total output is contributed by large corporations—roughly a score of which are capitalized at \$1,000,000 or more. Included in this group are two or three smelting companies and perhaps half a dozen other concerns that are active in other lines of manufacturing, but according to the

TABLE 10.—*Production of Specified Nonclay Refractories 1929 and 1932<sup>a</sup>*

Kind and State	1929			1932		
	Plants	Quantity	Value, Thousands of Dollars	Plants	Quantity	Value, Thousands of Dollars
Silica brick, total, thousands:	26	294,402	\$15,165	25	40,111	\$1,762
Pennsylvania .. . . .	14	213,060	10,453	13	27,778	1,184
Other states <sup>b</sup> ... . .	12	81,342	4,712	12	12,333	578
Magnesite and chrome brick, total, thousands <sup>c</sup> .....	4	17,399	5,631	3	3,167	845
Graphite crucibles, total, tons.	11	"	2,945	9	2,127	639
Pennsylvania .....	4	"	1,133	4	843	226
Other states <sup>d</sup> . . . . .	7	"	1,812	5	1,284	413
Refractory cement (nonclay), total, tons:	18	80,802	1,421	24	16,693	444
Pennsylvania.....	7	"	"	8	7,516	196
Other states <sup>e</sup> . . . . .	11	"	"	16	9,177	248
Others, including alumina and silicon carbide refractories, total tons:	20	"	6,989	29	"	2,414
Pennsylvania.....	6	"	"	9	"	472
Other states <sup>f</sup> . . . . .	14	"	"	20	"	1,942

<sup>a</sup> Compiled from U. S. Bureau of the Census reports.

<sup>b</sup> In 1929: Alabama, 1 plant; California, 1; Colorado, 2; Illinois, 2; Indiana, 1; Missouri, 1; Montana, 1; Ohio, 2; Utah, 1. In 1932, same except no plant in Missouri, and 2 in California.

<sup>c</sup> In 1932, Maryland, 1 plant; Pennsylvania, 2. In 1929 a third plant in Pennsylvania made magnesite brick but not chrome brick.

<sup>d</sup> In 1929: New Jersey, 3 plants; New York, 1; Connecticut, 1; Illinois, 1; Massachusetts, 1. In 1932: New Jersey, 3; Connecticut, 1; Massachusetts, 1.

<sup>e</sup> In 1929: Alabama, 1 plant; Colorado, 1; Illinois, 2; Indiana, 1; Massachusetts, 1; Montana, 1; New Jersey, 3; Ohio, 1. In 1932: Alabama, 2 plants; California, 1; Illinois, 1; Indiana, 1; Maryland, 1; Massachusetts, 1; Michigan, 1; Montana, 1; New Jersey, 4; New York, 2; Ohio, 1.

<sup>f</sup> In 1929: California, 1 plant; Illinois, 1; Kentucky, 1; Maryland, 1; Massachusetts, 2; Michigan, 1; Missouri, 1; New Jersey, 2; New York, 1; Ohio, 1; Washington, 1; West Virginia, 1. In 1932: Alabama, 1 plant; California, 2; Connecticut, 1; Kentucky, 1; Massachusetts, 2; Michigan, 2; New Jersey, 5; New York, 3; Ohio, 2; Washington, 1.

<sup>g</sup> Data not available.

reports of the Bureau of the Census 94.3 per cent of the value of the 1929 output of "fire-clay products" was supplied by 140 establishments reporting such products as their major products and 93.2 per cent of its aggregate value of "nonclay refractories" was reported by establishments that specialized in this field. A great many plants make only clay firebrick, occasionally adding certain allied fire-clay products, such as special shapes, refractory mortar, and plastic material; quite commonly, however, several brands of firebrick are made in the same plant, varying in composition or in process of manufacture (dry press, hand-molded, etc.). Silica brick are occasionally made in the same plant with fire-clay products but more often in a separate plant, although most of the production is furnished by companies that also produce clay brick. Magnesite and chrome brick are generally made in the same plant, whereas silicon carbide bricks are often made along with ordinary firebrick.

At least 20 companies operate two or more plants each, and three companies operate 10 or more plants each. Many plants that were once operated independently have been absorbed by other companies, and there is a marked tendency toward developing larger productive units. Despite the marked progress in horizontal consolidation the number and complicated system of individual brand names has not been correspondingly simplified. Firebrick, all rendering essentially the same type of service, are designated by probably 500 different brand names.

TABLE 11.—*Number of Establishments Producing Specified Products in the United States 1925-1934*

	1925	1926	1927	1928	1929	1930	1931	1932	1933	1934
Clay products:										
Clay firebrick, etc	238	230	214	213	198	196	183	178	170	181
High-alumina brick	10	14	13	16	17	14	10	12	17	25
Special shapes		40	39	41	53	63	66	73	64	69
Glasshouse refractories			14	13	13	14	16	16	15	16
Cement.....	38	42	40		48	55	53	51	61	57
Nonclay products:										
Silica brick.....	28	31	27	27	26	26	26	25	26	28
Magnesite brick	7	7	6	5	4	5	4	3	3	4
Chrome brick	4	4	4	4	3	4	4	3	3	4
Cement...	<sup>b</sup>	17	22	24	18	20	25	24	28	23
Other <sup>a</sup>	16	18	20	21	20	25	27	29	27	25

<sup>a</sup> Includes alumina and silicon carbide refractories.

<sup>b</sup> Nonclay cement included with clay cement in 1925.

#### IMPORTS AND EXPORTS

*Imports.*—The imports of firebrick and other refractories have been relatively small and in recent years have been growing smaller, as certain qualities of highly refractory fire-clay brick formerly imported from

Great Britain have been displaced by suitable domestic brick. Whereas both dead-burned magnesite and chrome ore are imported extensively into the United States, magnesite brick and chrome brick are made in this country in sufficient amount to supply the entire domestic demand with a surplus for export. Except for one or two substantial consignments of magnesite brick shipped from the U. S. S. R. in 1930 and 1931 imports of these high-priced brick have been negligible.

*Rates of Duty.*—Under the Tariff Act of 1930 (par. 201) magnesite brick are dutiable at  $\frac{3}{4}$  cent per pound and 10 per cent ad valorem, while chrome brick and all fire-clay brick and other firebrick not specially provided for are dutiable at 25 per cent of the foreign market value. Similar rates were provided in the act of 1922.

*Exports.*—Refractories, chiefly fire-clay brick, valued at one million dollars and more annually were exported from the United States before 1914. During the World War foreign orders increased enormously and continued on a greatly expanded scale until about 1921. In recent years the aggregate value of the exports of firebrick and refractory shapes has averaged around \$2,000,000 a year. While there has been some falling off in the exports of fire-clay brick, this loss has been made up to a considerable extent by larger exports of magnesite and chrome brick, the former with benefit of drawback or refund in the duties on the imported raw material.

TABLE 12.—*Imports of Refractory Brick for Consumption, 1923–1935<sup>a</sup>*

Year	Fire-clay Brick		Silica Brick		Magnesite Brick		Chrome Brick	
	Thou-sands	Value	Thou-sands	Value	Thou-sands <sup>b</sup>	Value	Thou-sands	Value
1923 . . .	8,117	\$184,211	c	c	0 5	\$735	12	\$607
1924 . . .	3,279	105,095	15	\$158	4.75	1,477		
1925 . . .	3,683	111,919	1	31	d	80		
1926 . . .	2,389	75,389			d	24		
1927 . . .	2,610	92,260	6	589				
1928 . . .	2,569	90,593			0.25	551		
1929 . . .	1,896	67,832			3.5	3,344	1	34
1930 . . .	1,537	54,113	30	694	1	715		
1931 . . .	714	25,034			252	49,399		
1932 . . .	430	15,573			2 5	450	3	477
1933 . . .	428	14,380			d	9		
1934 . . .	336	21,960			d	19		
1935 . . .	424	16,871		89	d	162		

<sup>a</sup> Not separately classified prior to Tariff Act of 1922.

<sup>b</sup> Reported in pounds; converted to thousands of brick by dividing by 9500.

<sup>c</sup> Included in firebrick not elsewhere specified.

<sup>d</sup> Less than 0.25 M.

TABLE 13.—*Exports of Refractories, 1919-1935*

Year	Fire-clay Brick		Other Refractory Brick		Refractory Shapes		Clay and Graphite Crucibles		Fire Clay	
	Thousands	Value	Thousands	Value	Short Tons	Value	Number	Value	Short Tons	Value
1919. . . .	51,252	\$2,747,512	"	"	"	"	"	"	37,486	\$262,501
1920. . . . .	82,570	4,200,266	"	"	"	"	"	"	54,125	393,177
1921. . . . .	35,853	2,314,911	"	"	"	"	"	"	23,666	177,979
1922. . . . .	30,961	1,407,175	5,577	\$ 236,096	5,071	\$ 174,504	355,155	\$ 92,273	24,391	169,664
1923. . . . .	42,552	1,711,792	5,577	471,866	13,785	620,744	463,265	132,228	50,055	303,675
1924. . . . .	24,056	709,650	12,732	1,126,678	15,765	753,762	548,893	96,001	41,704	312,676
1925. . . . .	22,938	643,729	14,083	1,315,124	13,259	679,399	408,249	91,699	44,037	330,371
1926. . . . .	25,620	708,068	14,003	1,378,577	13,861	693,741	618,735	149,466	47,737	348,302
1927. . . . .	23,500	874,254	13,465	1,338,238	20,170	811,664	558,000	108,857	48,106	392,327
1928. . . . .	29,576	815,447	16,718	1,454,695	29,244	1,273,593	351,855	110,012	60,138	494,241
1929. . . . .	30,604	847,745	21,736	1,922,650	42,239	1,862,726	692,769	124,466	76,561	588,770
1930. . . . .	25,387	880,872	10,951	1,121,382	40,311	1,672,024	488,999	103,802	62,660	519,788
1931. . . . .	17,526	331,691	4,739	490,729	7,969	475,925	350,152	65,613	45,314	329,112
1932. . . . .	7,551	464,727	2,951	253,610	4,268	285,563	"	"	22,086	228,073
1933. . . . .	8,928	530,745	4,122	381,335	5,611	392,842	"	"	32,432	264,595
1934. . . . .	16,526	1,004,846	6,250	718,073	9,177	717,329	"	"	36,052	303,424
1935. . . . .	16,037	1,092,520	5,734	623,977	10,349	742,772	"	"	49,949	380,604

\* Not listed separately.

Canada has always been the chief foreign market for American refractories, and the bulk of the remaining exports has gone to Latin American countries. Shipments to Europe have never amounted to more than a very small quantity, but there is a tendency for American metallurgists engaged in foreign work to specify American products with whose merits they are familiar, particularly in Africa, North and South America, and other localities where competitive European products are not favored by an overwhelming advantage in delivered cost.

#### PRICES

The prices of producers' goods are generally subject to much more violent fluctuations than the prices of consumers' goods. Refractories are raw materials in the sense that they are used up or worn out by the metallurgical industries, and as these industries are notoriously "prince or pauper" it follows that the consumption of refractories tends to ebb and flow between wide limits. Moreover, when steel plants are only partly occupied it frequently happens that idle furnaces are not repaired and that when repairs are necessary old brick and other used refractory materials from idle furnaces may be salvaged and used to reline a single active furnace, with the result that purchases of new refractories having thus been postponed, the peak demand at the start of a business upswing may be correspondingly exaggerated. Under these circumstances it is rather surprising to note that the prices of standard refractory materials and



products have generally been altered at rather infrequent intervals. Naturally there was a more or less violent distortion during and for a few years after the World War, and as the depression of the early 1930's became prolonged it likewise had some influence, but for several decades the price history of the refractories industry has been one of steady but gradual advances; reflecting, in part at least, improvement in the quality of the products and the more exacting specifications governing their production and properties. Gradual exhaustion of certain easily accessible deposits of fire clay also was a factor. As indicated by the reports of producers to the Geological Survey and Bureau of Mines the average annual value of fire clay which was generally around \$1 a ton at the beginning of the twentieth century had risen above \$1.50 by 1914 and after exceeding \$3 during the postwar boom has receded so reluctantly as to indicate a natural new level above \$2.50.

A fairly good idea of the trend of firebrick prices over a period of years is afforded by the data given in Table 14, showing the quoted prices of refractories since 1922. Price quotations for brick are for standard 9-in. brick; other sizes and shapes are subject to extras ranging from a small advance in the case of wedges and keys to a fairly large differential on soaps and splits and still higher extras on special shapes for which only a limited demand exists. However, this condition likewise has little effect upon national averages because sales are always dominantly in the standard series. In 1922, as reported from actual sales, the quotation for first-quality brick was \$43 per thousand. This price rose to \$45 early in 1923 and later in the same year fell to \$42. At the end of 1924 the price rose to \$43 and continued at this level, with minor exceptions, until the second quarter of 1931. During the second quarter of 1931 a downward revision of \$3 brought trade-journal quotations down to \$40, and later they dropped to \$35. In 1933, following the improvement in steelworks' demand, an upturn brought the quotation to \$45 in August.

Since a thousand fire-clay brick weigh around  $3\frac{1}{2}$  tons, the prices at eastern steelworks (including freight) generally run from \$7 to \$10 a thousand higher than the quotation f.o.b. kilns. Second-quality brick are nominally \$5 a thousand cheaper than first quality. Silica brick, which are currently quoted at \$54, or the same price as first quality fire-clay brick at Pennsylvania markets, are much higher in the West, being quoted recently at \$63 in Illinois and Indiana.

Until recently, magnesite brick have been stationary at \$65 per ton, and chrome brick have remained at \$45 per ton for several years, except for a time during the depths of the recent depression when they were quoted at \$61.50 and \$42.50, respectively. Unburned magnesite brick have been priced at \$10 per ton less than burned magnesite brick and unburned chrome brick at \$7.50 less than burned brick of similar kind. In 1933, dead-burned grain magnesite was quoted in trade journals at \$25

TABLE 14.—*Prices of Fire-clay Brick, Silica Brick, Chrome Brick and Magnesite Brick, 1922–1937<sup>a</sup>*  
F.o.b. Plants

Year	Fire-clay Brick, per Thousand <sup>b</sup>				Silica Brick, per Thousand				Chrome Brick, per Short Ton	Magnesite Brick, per Short Ton
	First Quality		Second Quality		Pennsylvania		Alabama			
	High	Low	High	Low	High	Low	High	Low		
1922.....	\$43@46	\$32@35	\$39@41	\$ 28	\$ 50	\$ 28	\$ 50	\$ 48	\$40@55	\$53@80
1923.....	45@48	40@46	41@43	36@41	45@47	42@44	53@55	48@50	50@52	65@68
1924.....	42@45	40@43	35@38	33@37	50	33	53	43	45@50	65@68
1925.....	43@46	43@46	36@40	35@40	42	40	52	45	40@50	65@68
1926.....	43@46	40@43	35@40	35@38	42	40	52	48	45@48	65
1927.....	43@45	40@43	35@40	35	43	40	52	49	45	65
1928.....	43@46	43@45	35@38	35@38	43	43	51	51	45	65
1929.....	43@46	43@46	35@38	35@38	43	43	51	51	45	65
1930.....	43@46	43	35@38	35	43	43	50@51		45	65
1931.....	43	40	35@38	35	43	40	50@51		45	65
1932.....	38	35	40	25@30	38	35	50	45	42 50@45	61.50@65
1933.....	45	35	40	30	45	35	55	45	42 50@45	61 50@65
1934.....	45	45	40	40	45	45	55	52	45	65
1935.....	45	45	40	40	45	45	52	50	45	65
1936.....	45	45	40	40	45	45	50	48	45	65
1937.....	54	54	49	49	54	54	54	54	49	69

<sup>a</sup> Engineering and Mining Journal quotations.

<sup>b</sup> Central Pennsylvania, Ohio, and Kentucky.

per short ton f.o.b. California mines until late in September, when it rose to \$28; the product from Chewelah, Wash., was quoted unchanged at \$22, f.o.b. plant, to which freight charges ranging from \$10 to \$15 a ton should be added to arrive at a price delivered at consuming points in Pennsylvania or Ohio. Chrome ore for refractory uses is somewhat cheaper than the higher grade material employed for making ferrochrome; Grecian refractory ore has recently been quoted at \$19 a gross ton c.i.f. Atlantic seaboard.

High-alumina brick containing 50 per cent  $\text{Al}_2\text{O}_3$  are currently quoted at \$91 per thousand, f.o.b. Missouri plants; 60 per cent  $\text{Al}_2\text{O}_3$ , \$100; 70 per cent  $\text{Al}_2\text{O}_3$ , \$141; and 80 per cent  $\text{Al}_2\text{O}_3$ , \$260. Fused-alumina brick are quoted at \$1.08 each; fused magnesia, at \$1 each; and silicon carbide, at \$1 each.

Zirconia brick are priced at \$1.10 each, kyanite brick range from 45¢ to \$1 each, and various other special brick and refractories are available over an exceedingly wide range of prices.

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## CHAPTER XXXVII

### SALT

By W. C. PHALEN,\* MEMBER A.I.M.E.

SALT is one of the primary human needs and the history of its production goes back to the beginnings of the human race. Because it is essential to human health, it has been a favorite basis for taxation and government monopoly and much of the fight for political and economic freedom has been at times tangled up with salt production, as in India in recent times. Necessity for salt led to surrender of the fierce head hunters of the hills to the Japanese in Formosa, and everywhere that salt production has been monopolized by a government it has been at once a source of power and a cause of unrest. Fortunately, in the United States the industry has always been a free one. Now salt has found so many other uses than as a food or food preservative, that it may well be considered the most essential raw material in modern chemical industry.

#### COMPOSITION AND PROPERTIES

Pure salt contains by weight 39.34 per cent sodium and 60.66 per cent chlorine. Expression of the purity of salines by radicals instead of by conventional combinations is the more accurate, though the latter is the custom in commercial analyses. Therefore, analyses of typical rock salt that is mined and marketed as such, and that also is one of the sources from which brine and evaporated salt are made in different parts of the United States, are expressed both ways in Table 1. These analyses indicate the purity of commercial rock salts. The brine derived from them should be of similar grade but saturated with respect to calcium sulphate. Care must be exercised in the evaporation process. For the food, table-salt and dairy-salt trades even small quantities of calcium sulphate and chlorides of magnesium and calcium are objectionable. The extensive use of chemicals for their removal, however, coupled with the low price of the marketed product, is prohibitive on account of the cost; therefore the degree of purification to which the brine may be subjected has to be closely figured. It may be said that the salt going into all branches of the food industry is of a very high degree of purity and here competition is one of the main regulating factors. In the salt industry the chemist has come into his own.

\* Syracuse, New York.

TABLE 1.—*Analyses of Rock Salt*

RADICALS IN PERCENTAGE OF MATERIAL SOLUBLE IN WATER									
Constituent	1	2	3	4	5	6	7	8	9
K.....	Tr.	Tr.	Tr.	Tr.	Tr.	0.1	Tr.	Tr.	Tr.
Na....	38.9	38.0	39.0	38.8	38.4	38.6	38.3	38.3	39.1
Ca....	0.2	1.0	Tr.	Tr.	0.8	0.4	0.8	0.4	Tr.
Mg....	0.1	0.1	0.2	0.3	Tr.	0.1	Tr.	0.3	0.1
Cl.....	59.6	59.7	59.3	59.8	60.0	60.0	60.0	60.2	59.9
SO <sub>4</sub> .....	1.2	1.2	0.5	1.0	0.8	0.8	0.8	0.6	0.9
	100.0	100.0	99.0	99.9	100.0	100.0	99.9	99.8	100.0
CONVENTIONAL COMBINATIONS									
Constituent	1	2	3	4	5	6	7	8	9
NaCl (by diff.)..	98.35	96.54	98.09	98.46	97.57	98.10	97.47	97.36	98.76
KCl.....	Tr.	Tr.	Tr.	Tr.	Tr.	0.19	Tr.	Tr.	Tr.
Na <sub>2</sub> SO <sub>4</sub> ....	0.48								0.75
CaSO <sub>4</sub> ....	0.68	1.70	Tr.	Tr.	1.14	1.14	1.14	0.85	Tr.
CaCl <sub>2</sub> ....		1.37			1.29	0.18	1.29	0.42	
MgSO <sub>4</sub> .....	0.49		0.62	1.25					0.49
MgCl <sub>2</sub> ..		0.39	0.29	0.19	Tr.	0.39	Tr.	1.17	
	100.00	100.00	99.00	99.90	100.00	100.00	99.90	99.80	100.00

Nos. 1, 2 and 3, Livingston County, New York. Nos. 7 and 8, Lyons, Kansas.

No. 4, near Detroit, Michigan.

No. 9, Louisiana.

Nos. 5 and 6, Kanopolis, Kansas.

TABLE 2.—*Analyses of Flake Grainer Salt, Used in Butter and Cheese*

Constituent	Composition, Per Cent		
	1	2	3
NaCl.....	99.69	99.90	99.91
CaSO <sub>4</sub> ....	0.26	0.06	
CaCl <sub>2</sub> .....	0.02		
MgCl <sub>2</sub> .....	0.02	0.03	
CaCO <sub>3</sub> .....	0.01	0.01	Tr.
Na <sub>2</sub> CO <sub>3</sub> .....			0.01
Na <sub>2</sub> SO <sub>4</sub> .....			0.08
	100.00	100.00	100.00

No. 1 was made from brine, treated with lime only. Nos. 2 and 3 were from brine, purified by treatment with lime and soda ash. The difference in purity is apparent.

As bearing on this point, the analyses in Table 2 of fine flake grainer salt made from chemically treated brine, produced by solution of rock salt, are given. Salt of the purity indicated in these three analyses may be made by proper chemical treatment of the corresponding brine from any of the rock salts of which the composition is given in Table 1.

An article on the practice of saltmaking and the character of the salt produced along the shore of Great Salt Lake, by T. B. Brighton and C. M. Dice,<sup>4</sup> is informative on certain phases of the business in that section of the West. These authors state that salt as produced at Great Salt Lake is treated by repeated crushings and screenings to produce hay salt, stock salt, ice cream salt, special stock, No. 1 salt, No. 2 salt, cattle block salt, table salt and dairy salt. They give an unusually complete series of analyses of the Great Salt Lake product (Table 3). This series of analyses well shows how the purity of the product increases as it passes through the refinery. The chief impurity is calcium sulphate.

TABLE 3.—*Analyses of Great Salt Lake Salt*<sup>a</sup>

Constituent	Composition, Per Cent									
	1	2	3	4	5	6	7	8	9	10
NaCl (by diff.)...	93.86	99.3	99.5	99.5	99.5	99.5	99.7	99.5	99.7	99.8
CaSO <sub>4</sub> ...	0.41	0.557	0.346	0.378	0.374	0.369	0.249	0.384	0.204	0.132
CaCl <sub>2</sub> ...										
MgCl <sub>2</sub> ...	1.91	0.043	0.043	0.043	0.077	0.034	0.026	0.043	0.034	0.034
Na <sub>2</sub> SO <sub>4</sub> ...	3.78	0.070	0.065	0.044	0.071	0.041	0.014	0.053	0.055	0.033
Insoluble	0.04	0.037	0.025	0.020	0.018	0.023	0.037	0.019	0.010	0.011
	100.00	100.007	99.979	99.985	100.040	99.972	100.026	99.999	100.003	100.010

<sup>a</sup> T. B. Brighton and C. M. Dice.

<sup>b</sup> All calcium is computed as sulphate and the remainder of the SO<sub>4</sub> as sodium sulphate.

No. 1, sample of salt, direct from ponds. No. 6, unscreened stock salt.

No. 2, salt from stockpile. No. 7, No. 1 salt.

No. 3, extra coarse kiln-dried. No. 8, No. 2 salt.

No. 4, coarse kiln-dried. No. 9, 40-mesh table salt.

No. 5, hay salt. No. 10, butter salt.

*Properties.*—Common or rock salt is known mineralogically as halite. It is rather brittle and has a conchoidal fracture. Its hardness is 2.5. Its specific gravity ranges from 2.1 to 2.6, that of pure crystals being about 2.17 (136 lb. to the cubic foot). Its index of refraction is 1.5442. It is highly diathermanous; and does not conduct electricity. It is transparent or translucent. Its luster is vitreous. It is plastic under pressure.

Halite is isometric in crystal form. As rock salt, its most common occurrence, it has a coarse, granular to compact structure. It often

<sup>4</sup> References are at the end of the chapter.

forms cubes, which are commonly distorted or joined in such way as to form hopper-shaped crystals. Masses with cubical cleavage are fairly common; much less common is the fibrous variety, said to be pseudomorphous after gypsum; also the stalactitic form.

When pure, halite is colorless, but this rarely occurs. Impurities, such as the more common metallic oxides of iron, impart to it shades of yellow or brown. The peculiar reddish tinge often observed in crystals produced by solar evaporation is considered by some to be due to a microscopic form of organic life. The blue mottlings sometimes observed are thought to be due to the presence of colloidal sodium, but the writer does not know that this has been confirmed. These different tints are responsible for its varying degrees of translucence.

Halite is readily soluble in water and its degree of solubility at 100° C. is but slightly greater than that at 0° C. This fact has a very important bearing on the cheap production of artificial brine, from which so much evaporated salt is made, and which enters so largely into the chemical industry. The characteristic saline taste of salt is known to all.

#### MODE OF OCCURRENCE AND ORIGIN

Salt occurs: (1) in solid form and (2) in solution. The common type in solid form is called rock salt. Salt in solution is generally referred to as brine, which may be of all degrees of saturation with respect to salt. Bitterns may be said to be a type of brine containing not only salt but commonly other chlorides together with bromides, iodides and sulphates, most often of the alkaline-earth group of metals. Generally bitterns are of a high degree of saturation, especially when formed under artificial conditions. In nature, some of them are rather weak.

*Rock Salt.*—Rock salt is the most important source of the prepared or commercial article and occurs in two widely different structural forms; namely, as (1) beds and (2) "salt structures," such as bosses, plugs, ridges or domes, as they are commonly called in Texas and Louisiana. Though the latter are associated with sedimentary rocks, they differ in form and origin from the common bedded or stratified type of rock salt.

The occurrence of rock-salt beds or lenses associated with the common types of sedimentary rocks of all geologic ages down to the present is well known. In addition to its stratified occurrence with sandstones, shales and less commonly limestones, it is interbedded with other saline minerals such as gypsum, anhydrite and the potash salines in various parts of the world, notably in northern Europe. Single beds or deposits, often hundreds of feet thick, are not uncommon, and often the areal extent of such deposits is immense, as in the great Permian Basin, extending from Kansas through Oklahoma into Texas and New Mexico, of which the area is officially reported to be about 100,000 square miles.

Great bosses, plugs or ridges of rock salt, differing structurally and genetically from the ordinary type of bedded deposit are known to occur in areas of sedimentary rocks throughout the world. DeGolyer, seeking a broad term to cover them all, refers to them as "salt structures." Such structures are found not only along or near the Gulf Coast in Texas and Louisiana, but also in Mexico, Germany, Rumania, Egypt, Persia, Russia, Spain, Algeria and Morocco. Possibly the occurrence of rock salt at Malagash, Nova Scotia, belongs here. As the occurrence along our own Gulf Coast is decidedly of the plug or boss type,\* they are appropriately referred to as salt domes.<sup>8</sup>

Most rock salt is intimately mixed with other salines, such as anhydrite, gypsum and with smaller quantities of the chlorides of calcium and magnesium. Ordinary commercial deposits of potash salts are generally mixtures of rock salt and potash salts in varying quantity. At Stassfurt, Germany, more than 30 such minerals with potash, lime and magnesia as well as soda as their basic element have been found. In addition to the saline minerals, rock salt may contain intimate admixtures of rock particles, such as shale, limestone, dolomite and sandstone and all gradations may occur from the nearly pure salt at one end of the series to practically barren rock at the other.

It is perhaps true to say, in the present state of our knowledge of rock-salt domes, speaking now of those in Louisiana and Texas, that not so great a percentage of impurities is present as in the bedded type of rock salt. It is known, however, that anhydrite and/or gypsum are abundant in dome salt and potash salts have been found at Markham, Texas. If the hypothesis be accepted that cap rock has resulted from solution of the rock salt of domes, it follows that the minerals composing the cap must have been present originally in the salt. M. A. Hanna and A. G. Wolf<sup>11</sup> list 22 distinct mineral species other than gypsum and anhydrite occurring in cap rock of Louisiana and Texas. Besides the minerals listed, certain rock types, such as sandstone, are found in the salt of our domestic domes. As time goes on and a more intimate knowledge is obtained of salt structure throughout the world, we may find that so far as admixed foreign matter is concerned, the two types are quite comparable.

*Salt in Solution.*—The most familiar example of salt in solution is sea or ocean water; but it also occurs as brine in the water of inland and coastal lakes or lagoons, generally without outlet, scattered through all parts of the world. Great Salt Lake, Utah, and the Dead Sea, in Palestine, are illustrations of the latter type. Salt is also found intermittently as thin crusts or in solution in shallow basins or playas, more especially in the arid regions of the earth. In the form of natural or connate brine, salt is widely distributed in the sedimentary rocks, in cavities or smaller

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\* See Fig. 1, p. 847.



pore spaces whence it comes to the surface as springs or is tapped in wells. In certain places, soils or porous glacial deposits are impregnated with brine. All these types of occurrence of brine salt are important in various parts of the earth, and will be referred to later under the head of distribution.

### ORIGIN

To outline the mode of occurrence of salt is an easy matter, but to explain satisfactorily the origin of even the comparatively simple bedded type of rock salt, such as occurs in New York, Ohio, Michigan and Ontario, is far from simple. Deposits of rock salt were formed originally by the gradual evaporation of sea water in bays or lagoons separated wholly or in part from the main oceans. The water of inland lakes or seas in different parts of the world is even now undergoing concentration by evaporation. The mineral matter of such enclosed bodies of salt water in general crystallizes out in the order in which the solution becomes saturated with the various salts, which is analogous to what takes place in the production of evaporated salt. This order depends partly on the relative quantities of the different salts or gases in solution and partly on their solubilities and conditions of pressure and temperature.

The order of precipitation of chemical deposits from sea water was demonstrated in 1849 by the Italian, J. Usiglio,<sup>2</sup> using water obtained from the Mediterranean Sea, several miles off-shore from Cette and from a depth of one meter. They show the order of precipitation to be: (1) iron hydroxide, (2) lime in the form of carbonate, (3) lime as sulphate, (4) the deposition of ordinary sodium chloride, and finally, (5) the accumulation of the soluble salts of potash and magnesium in the mother liquors. The deposits at Stassfurt, Germany, are the world's classic illustration of this order of precipitation, but exceptions to it are the rule rather than the exception. The commonest exception, of course, is the complete absence of the soluble potash and magnesium salts; but in places gypsum is present without the salt, and vice versa. Also, the enormous thickness of the two common salines must be explained. Precipitation that stopped when the gypsum had been laid down is easily understood; but the occurrence of salt not underlain by gypsum, or the occurrence of salt and gypsum in beds of which the thicknesses are out of all proportion to what theory calls for, require special theories of origin.

One such theory has been suggested by G. Bischof<sup>14</sup> and later by C. Ochsinius.<sup>9</sup> The latter's theory is sometimes referred to as the "bar theory." It supposes that evaporation, wholly or in part, took place in a sea or lagoon, isolated by a barrier from the open ocean. Occasionally new supplies of ocean water passed over or through one or more restricted channels in this barrier and thus ensured a continuing supply of saline material.

Other theories of salt deposition have been formulated. One of these has been called the "desert" theory of J. Walther.<sup>7</sup> According to this theory, salt deposits might originate from earlier or older occurrences of saline material, either present in the rocks as such or in the form of brines. Such salt would be secondary. Both theories involve arid conditions and a restricted area of deposition.

The Silurian salt deposits of North America were studied by A. W. Grabau,<sup>10</sup> and a voluminous literature resulted. An interesting summary of nine theories as to the origin of salt in the Michigan, Ontario and the New York and Pennsylvania basins are given by H. L. Alling,<sup>1</sup> and there are outlined objections and discussions relating to the different theories of salt deposition in these basins with special emphasis on Grabau's view. The bibliography accompanying the descriptions will enable the student to pursue the subject as far as he desires.<sup>1,10</sup>

An entirely different explanation is forthcoming with reference to the "salt structures" of the world, more especially the domes of the Gulf Coast States. The theory of the formation of the latter domes by the plastic flowage of sedimentary salt under differential pressure has now come to be generally accepted by men that have studied the subject most intensively. Torsion balance data indicate that the base of the salt core of these domes lies in some places at depths of 17,000 to 20,000 ft., as in the Houston, Texas, district.<sup>8,9</sup> The age of the salt is as old as the earliest Lower Cretaceous and the salt plugs are now found penetrating all the younger rocks.

#### DISTRIBUTION OF DEPOSITS

Salt is widely distributed. In many countries, it occurs in beds of sufficient thickness and areal extent to constitute true rock masses. A list of localities where it is found in all the forms enumerated on the preceding pages would include practically every political division of the world.

*United States.*—In the United States, extensive deposits of rock salt are found in central and western New York. The southern dip of the beds of New York carries the salt into Pennsylvania, but along the northern border of the latter, as well as in the Pittsburgh district, the deposit is buried deeply. Rock salt is also found in Ohio, Michigan, Virginia, Louisiana, Mississippi,\* Arkansas, Kansas, Oklahoma, Texas, New Mexico, Colorado, Nevada, Utah, Arizona, California, Idaho, Wyoming, and perhaps in other states. Although rock salt, the main dependence of the salt industry, is widely scattered over the United States, it is lacking over extensive areas. It is not present for example, in the New England States, where consumption is large. This enables foreign salt to compete successfully along the coast, especially in the fishing industry. Excepting a deposit of rock salt in southwestern Virginia, which forms the basis of an

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\* Since this chapter was written, salt has been discovered in Mississippi.

alkali industry, no rock salt is known to occur south of Ohio River and east of Mississippi River, except locally in Louisiana. In the Mississippi Valley, north of Louisiana and Arkansas, there is a vast area where no rock salt occurs. In the Northwest, but little is known and there is none in Washington and Oregon. Doubtless study and exploration will reveal its occurrence in some of this territory. In many of the states mentioned, rock salt is the basis of an important production of the commercial product as well as chemicals derived from it.

Sea water is the basis of an important solar salt industry along the California coast, especially along the shores of San Francisco Bay, near Los Angeles and San Diego, and the water of Great Salt Lake is evaporated similarly along its shores. Playa salt is of local importance in some of the more arid western states. Connate brine is now the source of a considerable salt industry along the Ohio River in Ohio and West Virginia; and along the Kanawha River in West Virginia. In the Saginaw Valley, Michigan, the salt industry based on connate brine was formerly a large one and it is still considerable. It marked the beginning of an important chemical industry. In Oklahoma, natural brines have been utilized in the vicinity of Tulsa. Natural brines stored in the glacial gravels and soil near Syracuse, N. Y., and which in early days emerged as springs, have formed the basis of a salt industry in the past and determined the location of the present chemical industry.

*Canada.*—There are notable deposits of rock salt in several of the provinces of Canada. In Nova Scotia a rock-salt industry has grown up in the Malagash district. Extensive deposits are known in New Brunswick; and in Ontario, large quantities of evaporated salt as well as other sodium chemicals are made from the important and extensive deposits in the southern part of the province. Saline deposits are known in northern Manitoba and rock-salt deposits in the McMurray district of Alberta. Rock salt is reported in drill holes near Unity, Saskatchewan, and saline springs as well as rock salt are reported at a few places in British Columbia.<sup>5</sup>

*Mexico.*—Deposits of rock salt occur at numerous localities in the States of Zacatecas, Coahuila, Sonora and others, but have not been worked to any great extent. Considerable quantities of salt are obtained from sea water on the Gulf of Mexico and that of Lower California, notably at Carmen Island, Lower California; and at Cuyutlan, State of Colima. This solar lagoon deposit and another at Palomas, northern Mexico, between Sierra Mojada and Escalon, have been worked by an English company. In the State of Puebla there are numerous saline springs, and brine from shallow wells is evaporated by solar process at Chila and vicinity, 30 miles south of Matamoras. Salt deposits in one form or another are found in more than 20 states and more than 90,000 metric tons per annum are now being produced.

*Central America.*—In Guatemala, rock salt is mined in the Departments of Huehuetenango and Verapaz. In Costa Rica, a concession was granted some years ago for salt pans over an area of about 250 acres on the Gulf of Nicoya. A condition was that production should not be less than 250 metric tons yearly. In Panama, salt is produced by the solar evaporation of sea water at Aguadulce, Province of Coclé, and elsewhere.

*West Indies.*—The production of solar salt in the West Indies, which at one time was an important industry, has decreased considerably since the United States began to produce enough salt to meet its own requirements. It was produced in quite primitive fashion. At present the only considerable output is in the Bahama group and in the Turks and Caicos Islands, which form a separately administered group farther south. These are all parts of the British Empire. Among the Netherlands West Indies, salt is produced from sea water on the Islands of Curaçao, Bonaire and St. Martin. At Philipsburg, on the last-named island, salts of magnesium and potash have been separated from the crude salt. Large deposits of rock salt occur in the Dominican Republic.

*South America.*<sup>16</sup>—In Argentina, salt is widely distributed in salt lakes, where it has concentrated naturally through solar evaporation under arid conditions. These deposits supply local needs, but no large works have yet been built except in the southern part of Buenos Aires Province, where an evaporating plant on San Blas Bay recovers salt from brine pumped from a near-by lake. Salt is collected during the dry season along the shores of lakes at Salinas Grandes, on Valdez Peninsula, Province of Chubut. A railroad built to the coast facilitates shipments. At San Luis, considerable salt comes from Lake Behedero, about 25 miles south of the city. There are several important salt lakes within easy reach of the railroads, which might be used as sources of salt, notably in the Sierras de Córdoba, in Los Andes, Jujuy, Salta, Buenos Aires and San Juan.

In Bolivia, salt occurs over a large area in the Coipasa district, west of the railway between Uyuni and Oruro. It has been worked at Garcimendoza.

Although salt is made in some quantity in Brazil by the solar evaporation of sea water, much salt is imported. The chief salines are in the states of Rio Grande do Norte, Alagoas, Sergipe, Ceará, Piauí and Rio de Janeiro. Small salt pans occur in the valley of São Francisco in the State of Bahia. Rock salt is said to have been mined in Mato Grosso and Goiás.

Chile produces enough salt for its own requirements, except for a small importation of refined salts. Large salt pans known as "salares" occur throughout the arid districts of northern Chile, notably the Salar Grande, in the southern part of the Province of Tarapacá. The Salar Grande covers about 80,000 acres and contains practically pure salt. Its

thickness is unknown but a shaft 82 ft. deep failed to penetrate the deposit. A Salar at Lagunas supplies three crops of solar salt yearly.

In Colombia, salt is a government monopoly. It is made partly under direct control of the state and partly by authorized contractors, whose operations are open to official inspection. It is obtained as rock salt or in brine form from deposits east of Magdalena River in the departments of Cundinamarca and Boyaca. It is also obtained from sea water in salinas along the Caribbean Coast. Smaller quantities for local needs are supplied from brine springs in the departments of Antioquia, Caldas, Cauca, Narino and Huila. The principal deposit of rock salt is at Zipaquira, Cundinamarca, on the railroad north of Bogota. The deposit is estimated to contain 1,000,000,000 tons of rock salt. Its geologic age is not stated.

Salt is a government monopoly in Ecuador and enough is produced for local needs. It is produced chiefly in the Santa Elena district from the solar evaporation of brine; also at Mira, province of Carchi, from saline earths and from brine springs at Salinas.

In Peru, the salt industry is a government monopoly administered by a salt company which itself works most of the sources but buys also from certain privately owned salinas, thus entirely controlling distribution. The salt is obtained principally from sea water, but some comes from rock-salt deposits and from brine springs. The salt made from sea water is of excellent quality and comes from Huacho, 100 miles north of Lima; from Casma; and from the Salina del Cerro, in the Sechura district, province of Piura. The brine springs of Guadalupito are about six miles from the port of Chimbote. The rock-salt deposits are considered to be of Permo-Triassic age. At San Blas, 18 miles west of Cerro de Pasco, rock salt is found. At Cachi Cuyao, near Izcuchaca, Province of Huancavelica, the best of the rock salt averages 40 ft. At Atacocha, 18 miles north of Ayacucho, the outcrop extends 2500 ft. and averages 150 ft. in thickness, with reserves estimated at 6,000,000 tons. Another deposit at Cachi-huancaray, 30 miles east of Andahuaylas, is estimated to contain 1,200,000 tons of salt.

In Venezuela, the salt industry is also a government monopoly. The product is obtained from salt pans formed by the evaporation of sea water in coastal lagoons. The principal deposit is on the Araya Peninsula. Other important producing districts are on the island of Coche and in the States of Falcon and Zulia.

*Europe.*—Rock salt is widely distributed in Europe, as are also the chemical industries based on it. It is found, for example, in the United Kingdom (England and Northern Ireland), Austria, Belgium, Czechoslovakia, France, Germany, Italy, Yugoslavia, Netherlands, Poland, Rumania, Soviet Russia, Spain and Switzerland. In these countries the commercial article is produced directly from the rock salt mined or from brines derived from the rock salt in place. The solar evaporation of sea

water is an important industry along the Mediterranean coast and elsewhere where climatic conditions permit, as in Bulgaria, France, Greece, Italy, Portugal, Soviet Russia, Spain and Turkey. In certain of the countries named—Austria, Czechoslovakia, Greece and Rumania—the salt industry is a government monopoly; i.e., the salt is made and sold under government supervision and taxes are collected on it.

*Asia.*—Though rock salt is known and worked in certain countries in Asia, it is probably true to say that the greater part of the commercial salt is produced by the solar evaporation of sea water, brine from springs or wells, and salt lakes. Rock salt is found in Arabia, China, India, Palestine, Persia, Asiatic Russia and Asiatic Turkey. Solar salt is made in Arabia, Ceylon, China, Cyprus, Netherlands Indies, Hong Kong, India, Indo China, Palestine, Weihaiwei Territory, Japan including Chosen (Korea) and Taiwan (Formosa), Philippine Islands, Syria, Siam and Asiatic Russia. In many of these countries, the industry is controlled by the government and is subject to tax. The actual manufacture may be in private hands, sometimes under government agents. Such monopolies exist in China, Cyprus, India, Indo China, Japan, Netherlands Indies, Palestine, Asiatic Russia and Turkey in Asia.

*Africa.*—Rock salt is known to occur in Southwest African territory belonging to the British Empire, also in Algeria, Angola, Eritrea, Morocco and Tunis. In nearly all parts of the continent, salt occurs in the water of lakes, springs and in great salt plains or playas covered by water parts of the year and dry during the arid season, when the salt is harvested. Abundant harvests are gathered from solar evaporated sea water along the coast. Perhaps the latter is the most abundant source. Salt resulting from solar evaporation is gathered in the following parts of the British Empire in Africa: Anglo-Egyptian Sudan, Gold Coast, Kenya, Mauritius, Nigeria, Rhodesia, Somaliland, Southwest African territory, Tanganyika, Uganda, and in the Union of South Africa. In the latter, artificial heat is employed in producing some of the salt. Solar evaporation is also employed to obtain salt in Algeria, Angola, Belgian Congo, Egypt, Eritrea, Italian Libya, Madagascar, Morocco, French and Italian Somaliland and Tunis. Salt occurs in the northern part of Abyssinia in the province of Tigre as well as in the great salt plain. The chief centers are Makalle and Dessie.

*Oceania.*—In Australia, quantities of salt are produced by evaporation of the saline waters of lakes, also from sea water. A large alkali plant now being built at Port Adelaide, South Australia, will use solar salt. In New Zealand, sea water is the main source of salt.

#### POLITICAL AND COMMERCIAL CONTROL

Control of the salt industry is of relatively small importance, because, generally speaking, each country is able to take care of its own pressing needs. Exchange of salt between the nations depends on other factors

than on conditions of control. Thus, although the United States leads in the output of salt among the nations and could produce far more than it needs, some foreign salt is imported. Salt from Spain, the United Kingdom, also from Jamaica, Turks Islands, the Netherlands West Indies, Nova Scotia, and even from northern Africa, competes freely along the Atlantic seaboard. A short distance from the coast, this influence is not felt. In recent years from 50 to 60 per cent of the salt imported into the United States came from Spain, and some of it, at least, is reported to reach Pacific Coast ports for use in the chemical industry. Generally such imported salt comes in very cheaply as ballast and this is the reason why it can compete. The fact that some foreign countries exercise a monopolistic control over their domestic production of salt makes no difference in world trade in this commodity. The monopolies are maintained for the revenue.

The tariff act that became effective in the United States on June 18, 1930, provides that sodium chloride or salt in bags, sacks, barrels or other packages shall be dutiable at 11¢ per 100 lb.; and in bulk at 7¢ per 100 lb. The following special provisions relate to salt:

1. Imported salt for curing fish: Imported salt in bond may be used in curing fish taken by vessels licensed to engage in the fisheries, and in curing fish on the shores of the navigable waters of the United States, whether such fish are taken by licensed or unlicensed vessels,

TABLE 4.—*Salt Imports and Exports*<sup>a</sup>

IMPORTATIONS								
Year	For Curing Fish		In Bags, Barrels and Other Packages		In Bulk		Total	
	Short Tons	Value	Short Tons	Value	Short Tons	Value	Short Tons	Value
1931	16,354	\$27,042	1,465	\$21,343	15,397	\$36,126	33,216	\$84,511
1932	11,110	14,034	1,728	21,056	15,180	30,955	28,018	66,045
1933	17,424	25,510	1,803	16,566	10,901	27,231	30,128	69,307
1934	31,734	56,662	2,296	19,334	19,094	44,524	53,124	120,520
1935	26,990	53,623	1,960	15,590	22,295	38,558	51,245	107,771

EXPORTS		
Year	Short Tons	Value
1931	98,710	\$775,490
1932	63,581	478,435
1933	105,178	626,694
1934	105,365	615,724
1935	112,212	549,522

<sup>a</sup> Figures from U. S. Bureau of Mines.

and upon proof that the salt has been used for either of such purposes, the duties on the same shall be remitted.

2. Exportation of meats cured with imported salt: Upon the exportation of meats, whether packed or smoked, which have been cured in the United States with imported salt, there shall be refunded, upon satisfactory proof that such meats have been cured with imported salt, the duties paid on the salt so used in curing such exported meats in amounts not less than \$100.

Table 4 gives the imports and exports of salt in recent years. Exports usually more than offset imports, as the table shows, so that the balance of trade is in favor of the United States, both in quantity and value. In 1935, imports were 0.6 per cent and exports 1.4 per cent of domestic production.

### PRODUCTION AND CONSUMPTION

As elsewhere, the history of production and consumption of salt in this country goes back to the beginning of settlement. This has followed from its use in the preparation and preservation of food. From such simple beginnings, its uses have expanded along many lines until the

TABLE 5.—*Production, Consumption and Value of Salt in the United States<sup>a</sup>*

PRODUCTION AND VALUE						
Year	Short Tons				Value	
	Evaporated	In Brine	Rock Salt Mined	Total	Total	Average per Ton
1931	2,203,690	3,300,210	1,854,170	7,358,070	\$21,541,012	\$2 93
1932	2,053,421	2,769,821	1,584,731	6,407,973	19,938,830	3 11
1933	2,358,954	3,461,026	1,784,992	7,604,972	22,318,086	2.93
1934	2,281,453	3,417,439	1,913,182	7,612,074	22,850,797	3.00
1935	2,345,000	3,860,000	1,800,000	8,005,000	22,226,000	2.78

### SALT MARKETED OR USED BY PRODUCERS IN 1935

Method of Production	Short Tons	Value	
		Total	Average
Evaporated in: open pans or grainers. . . . .	470,000	\$ 3,780,000	\$8.04
Vacuum pans. . . . .	1,400,000	8,845,000	6.32
Solar process . . . . .	347,000	1,300,000	3.75
Pressed blocks from evaporated salt. . . . .	128,000	925,000	7.23
Rock. . . . .	1,773,000	5,620,000	3.17
Pressed blocks from rock salt. . . . .	27,000	146,000	5.41
Salt in brine, sold or used as such. . . . .	3,860,000	1,610,000	0.42
	8,005,000	\$22,226,000	2.78

<sup>a</sup> Figures from the U. S. Bureau of Mines.



domestic chemical industries alone consume more than one-half the entire output. In Europe, presumably, conditions exist much as in this country. In the rest of the world, with the exception of Canada and Japan, the chemical requirements for salt have not kept pace with those in either

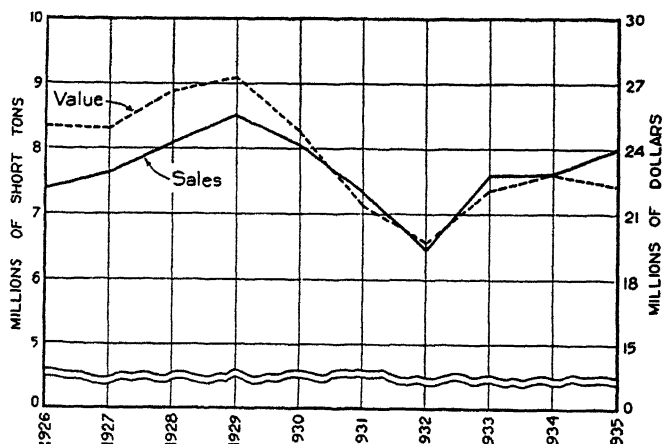


FIG. 1.—QUANTITY AND VALUE OF SALT SOLD OR USED BY PRODUCERS IN THE UNITED STATES, 1926–1935.

Minerals Yearbook, U. S. Bureau of Mines, 1936.

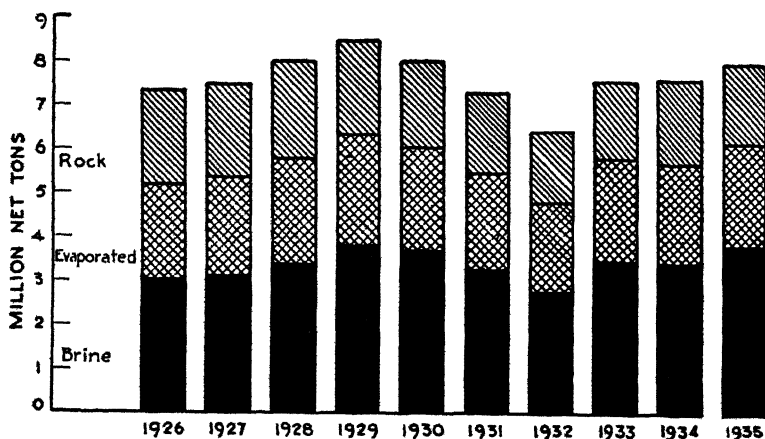


FIG. 2.—TRENDS IN QUANTITY OF ROCK SALT, EVAPORATED SALT, AND BRINE IN TERMS OF SALT CONTENT SOLD OR USED BY PRODUCERS IN THE UNITED STATES, 1926–1935.

Minerals Yearbook, U. S. Bureau of Mines, 1936.

the United States or Europe, but consumption for human use has steadily advanced proportionally to the increase in population.

In the location of salt-producing plants, a plentiful supply of raw material is the first essential and the availability of saturated brine is important. For such a low-priced commodity a good market close at hand is necessary. Other favorable factors are water for plant and cooling

purposes, as many competing railroads as possible, water transportation if possible, cheap fuel and a good supply of common labor. The manufacture of other products such as bromine, bromides, calcium chloride, magnesium metal and alloys, magnesium chloride and other chemicals, does not appear to be essential in this country.

The marketed production of salt in the United States is shown in Table 5. The values are f.o.b. mine or plant and do not include cost of containers. The curves of Figs. 1 and 2, which are for the latest 10-yr. period, show normal increase in quantity to 1929 with rapid decrease since that year to 1932 both in quantity and total value. Beginning in 1933, the industry has been on the upgrade.

By evaporated salt is meant that produced in single and multiple-effect vacuum pans, in grainers, open pans or in ponds or lagoons exposed to the sun's rays. The so-called brine salt is converted directly in large measure to alkali, hydrochloric acid, metallic sodium or chlorine and does not appear in the trade unconverted.

The leading place in the entire salt industry has been occupied for some years by Michigan, with New York second and Ohio third, and these three states supplied 70 per cent of the total salt sold and used by producers in 1935. There is a tendency for the number of plants to decrease as more efficient methods of production are introduced and capital expenditures grow larger. Only 74 plants were reported in operation in 1935, or 73 per cent of the number (102) in operation 15 years ago.

When an intensive study of the salt industry was made by the writer some years ago, it was found that plant capacity exceeded demand by one-third, a condition that later was found to be true by the U. S. Census Bureau. In view of the present business situation, it is more than likely that combined output is well below maximum capacity based on a demand requiring full running time.

*Local Elements Influencing the Domestic Industry.*<sup>19</sup>—Very few producing districts have a combination of all the advantages desired by a salt plant but certain districts either have or have had a combination of outstanding advantages. In the Saginaw Valley, Michigan, especially in the early days, the refuse from the lumber industry afforded fuel for evaporating the brines. This fuel cost nothing because it solved the troublesome problem of disposing of the waste from the saw mills. From the natural brine of this district, were made salt, bromine, bromides, calcium chloride, magnesium salts and other products. All these factors made possible such cheap salt that the western market for New York salt was completely destroyed years ago. The effect of the lumber business on salt production in Michigan has now become chiefly of historical interest.

The situation on the Ohio River, near Pomeroy, Ohio, and Mason, West Virginia, is similar to that which formerly existed in the Saginaw Valley. Here coal mines are close to the salt-evaporating plants and the

slack would become a nuisance if it could not be used in some useful way. Here also there is cheap water transportation on the Ohio River. In this district, bromine and calcium chloride are made. On the Kanawha River, West Virginia, the situation is similar to that on the Ohio River but natural gas is used as fuel. The salt produced along the Ohio and Kanawha Rivers also has a natural market of large area. Caustic soda and chlorine are made from West Virginia natural brine.

The industry in Saginaw Valley and on the Ohio and Kanawha Rivers is based on a supply of rather weak natural brine and never could have survived without the outstanding advantages already mentioned. The industries based on this natural brine have survived over a long period of years, individual salt plants based on it have been generally, but more particularly in the South, on a small scale.

*World Production*<sup>17</sup>.—The figures of world production of salt are available for as late as 1933, but output from certain countries is omitted, so that the figures are incomplete. However, the countries not accounted for are relatively unimportant, so that the information on world production as published by the U. S. Bureau of Mines must be very close to the facts (Table 6).

TABLE 6.—*World Output of Salt*<sup>a</sup>  
METRIC TONS

Continent	1929	1930	1931	1932	1933
North America ..	8,218,472	7,721,596	7,069,442	6,205,620	7,330,147
South America	337,110	288,497	245,378	287,786	314,754
Europe . . . .	10,786,123	10,471,403	9,869,014	9,852,637	9,853,497
Asia . . . . .	5,612,753	5,290,721	5,291,717	4,756,636	4,193,447
Africa .	647,091	763,238	597,372	680,817	629,115
Oceania . . .	135,684	117,829	123,769	113,842	109,527
Total . . . .	25,737,233	24,653,284	23,196,692	21,897,338	22,430,487
Production of United States . . . .	7,750,546	7,306,827	6,675,094	5,813,185	6,899,078
Per cent of total produced by United States . . . .	30.1	29.6	28.8	26.5	30.8

<sup>a</sup> Figures from U. S. Bureau of Mines.

The United States leads among the nations. In recent years Germany has ranked second, according to the official statistics, but in 1933 the United Kingdom forged into second place. China and India are huge consumers, because of their immense populations. World statistics show in general the same trends in recent years as do the domestic figures; i.e., maximum tonnages in 1929 gradually tapering off through 1932 and again rising in 1933.

## PROSPECTING, EXPLORATION AND MINING

Salt, a highly soluble mineral, does not appear at the surface except in arid regions. In the West, for example, outcrop areas of salt occur in Utah, Nevada, New Mexico, California, and possibly in other states. In areas of abundant rainfall, any salt remaining is usually buried so deep below the surface that it has been found only by drilling. The history of the discovery of salt in many places shows that it has been found incidentally—most often, perhaps, in the endeavor to find other substances, chiefly water, oil and gas. This has been generally true among the salt domes of Louisiana and Texas, but is not always so everywhere, however, as the presence of brine springs in the vicinity or in remote locations, but with an indicated geological relationship, has led to the discovery of strategically located rock-salt deposits. A single instance will be sufficient to indicate how geological reasoning led to the discovery of rock salt in Onondaga County, New York, and to the important chemical industry based on it.

The salt industry in New York State is the oldest in the United States. Salt was made by the Indians from brine springs, the most important of which, near Syracuse, Onondaga County, were noted by missionaries about the middle of the seventeenth century. It had long been believed by the geologists of the New York State Survey that the Onondaga (Syracuse) salt springs were supplied by the leaching of salt beds to the south, perhaps somewhere along the course of Onondaga Creek. As early as 1820, explorations with the drill were begun on the Onondaga Indian Reservation in search of these beds, but they resulted in failure. Other failures ensued, but the truth of the geologists' reasoning was demonstrated later and in a signal manner.

In 1888, the Solvay Process Co., in searching for a more abundant and cheaper source of brine supply than the partly concentrated brine springs offered for its soda-ash plant at Syracuse, began to sink wells in the valley of Onondaga Creek to the south of the city. Other fruitless wells were drilled at Jamesville and Cedarvale. All these early attempts, though based on sound geological reasoning, were failures, because the salt had been leached in the course of the ages. Finally an attempt was made at the very head of the valley and the drill encountered a satisfactory bed of salt at a depth of about 1200 ft. At last a location had been found where the covering of rock was impervious and thick enough to protect the underlying soluble salt from leaching.

To sum up the matter, it may be said that rock salt is prospected for by drilling, making use of either ordinary cable or rotary tools and coring where the desired data must be quantitative. The ordinary geophysical methods applicable in the location of salt domes in the Gulf Coast area are really means of locating salt. Where the salt, or brine, occurs in

low-dipping or flat-lying beds, resistivity methods seem to be indicated but experience in this last direction appears to be limited.

Salt is mined to some extent like the bulk of other minerals; but by far the greatest part of it is produced by dissolving it in water underground. The more or less completely saturated brine is lifted to the surface and the succeeding steps leading to the finished product may be considered a metallurgical process of refinement or a chemical process but less appropriately a manufacturing one, though it is commonly referred to by that term.

Ordinary water at air temperature is allowed to enter the well by gravity and the brine formed is lifted to the surface (a type of hydraulic mining), settled, purified and evaporated. It is obvious that the weight of the water column will lift the brine column a certain distance, depending on their relative specific gravities; 100 parts of water at ordinary temperatures dissolve a little more than 35 parts of salt by weight, giving a solution that contains about 26.5 per cent of salt and having a specific gravity of about 1.2, therefore a column of water of certain height will lift a column of saturated brine to  $\frac{5}{6}$  of that height. The brine is lifted the remaining distance (the actually applied lift) by compressed air—an air-lift, as it is usually called. Under certain circumstances where it is feasible, direct pressure may be used to lift the brine the required distance, and hydrostatic pressure is probably the cheaper means. Pumps may also be employed.

These operations are carried out in a system of concentric pipes. The water may be introduced through a casing. The brine accumulates at the bottom of the well and is forced part way out through the tubing. Within the tubing is placed the air line that accomplishes the final lift.

The literature on rock-salt mining is becoming a little more abundant; a few years ago almost no descriptions were in print on this important phase of mining. There is no exact agreement as to details of the methods used in rock-salt mining, as each district presents its own problems. An excellent description of the methods employed in an important mine in New York State, the Retsof mine, is given by E. F. LaVigne.<sup>12</sup> Operations along the Gulf Coast at Jefferson Island, Louisiana, and at Grand Saline, Texas, have been described by W. M. Weigel<sup>22</sup> and at the latter place also by H. B. Cooley.<sup>6</sup>

The work at Retsof, Livingston County, N. Y., will be taken as a type of a modern rock-salt mining operation. The new shaft, sunk in 1922, is elliptical in cross section, measuring 9 ft. wide by 28 ft. long. It is lined throughout with concrete and is 1063 ft. deep. It was designed for a capacity of 3000 tons in 8 hr. The salt bed now mined is 9 to 10 ft. thick. The panel system of mining is used. Each panel is 1330 ft. long and 375 ft. wide on centers. Double gangways are driven, one of these being an air way. Off the gangway, rooms 50 ft. wide are opened on 100-ft.

centers, leaving 50-ft. pillars for roof support. Crosscuts are driven 50 ft. wide; pillars are 50 ft. wide by 112 ft. long. The mining operations may be classified as: (1) undercutting, (2) drilling, (3) blasting, (4) underground loading, (5) haulage. Sixty-three per cent of the salt is extracted and the rest is left in pillars. There is practically no loss. Details of these different operations cannot be given, as they are the subject of a recent paper<sup>12</sup> but they follow closely the best practice in up-to-date soft-coal mining. Salt, being tougher than coal, the speeds and feeds of the machine are reduced and the undercutter bits are made of high-grade tool steel. No timber is used and there is no gas to contend with.

In the extraction of brine salt by water alone, solution of the rock salt underground may be considered a type of mining, and a most efficient type, because if properly done there is little salt left in the ground within reasonable distance of the circulating water.

#### PREPARATION FOR MARKET

The different kinds of salt marketed are: vacuum-pan, grainer or open-pan, solar and rock salt. Pressed blocks of salt may be made from both evaporated and rock salt. The first three types mentioned come under the general classification of evaporated salt.

The brine coming from the wells contains soluble impurities, chiefly hydrogen sulphide gas, iron compounds probably as carbonate, and calcium and magnesium salts. For the purest grades of salt used in the food industries and for special purposes, these are largely removed by chemical means. The brine may be aerated, treated with lime or lime and soda ash, and possibly alum, and allowed to settle a few days. Some manufacturers chlorinate the raw brine for the removal of sulphides and oxidation of the iron. The brine is then aerated to remove the excess of chlorine and the chemicals added. In the manufacture of vacuum-pan or grainer salt for the most exacting trade, the brine may be purified by the lime-soda-ash process and for this trade, largely the dairy, some manufacturers consider the cubical grained or granulated salt as good as the flake grainer salt.

The addition of such chemicals as lime and soda ash should be under close chemical supervision. Chemical dosage and control may be based on the pH value or, preferably, on actual chemical analysis or on alkalinity of the purified product. When purification takes place in the open, the brine surface always gathers some dust or fly ash and this must be avoided as far as possible.

Ingenious chemical methods based on sound principles have been suggested to prevent scale formation in vacuum-pan operations, such as taking advantage of the calcium chloride naturally present in the brine. In the manufacture of vacuum-pan salt, trisodium phosphate is sometimes added to the brine either in the pan or before it goes into the pan.

Doubtless other chemical devices are used, which have not come to the writer's attention. The next step in the preparation for market is the actual crystallization of the salt from the brine by the methods already stated.

The vacuum pans used in making granulated salt are generally of the vertical type. They consist of long vertical tubes, the ends of which are expanded into two plates, which form diaphragms across a cylindrical shell near the ends, the space between being the steam chest. There may be one or more in series, and they are referred to as single "effects," double or triple "effects," depending on their number. Those used in salt evaporation generally have deep cone bottoms. Usually they work with low steam pressure (5 lb.) and are run on a vacuum of 28 in. Usually such evaporators have large central downtakes and are fitted with special propellers for better circulation of brine.

The salt is recovered in closed filters either periodically or by a continuous vacuum process or by bucket elevators. Without proper pretreatment of the brine, scales form and evaporation is retarded. Scaling, unless prevented chemically, must be resorted to daily by mechanical means. Some brines foam and a large vapor space is required. Evaporators are generally built with cast-iron shells and with copper or brass tubes, but iron tubes are used. Very large units are the order of the day, even reaching a capacity of 1000 tons daily.

Vacuum-pan salt is made up of fine, lustrous grains, and by virtue of its fineness and purity is particularly suitable for the table, for canning foods and in the manufacture of patented prepared flours for cake, pancakes, doughnuts, etc., which have become so popular in recent years. It is better known to the trade as granulated salt. Each producer has his own classification, but in general table salt is about 30 mesh. Still finer grades, say through 30 and on 50-mesh screen, is commonly referred to as salt flour and is used in the patented flours mentioned. The grades coarser than table salt are largely consumed in the canning industry. Granulated salt may also go into the different dairy products.

To render certain grades of table salt moistureproof, so as to make them run as freely as possible, a small quantity, usually not more than 1 per cent of some nonhygroscopic substance like magnesium or calcium carbonate or phosphate is used to coat the grains. The product is the familiar "shaker" or free-running salt. Some salt is now mixed with a minute quantity of potassium iodide to make iodized table salt. The table grades for household use are packed and shipped in the familiar pasteboard cartons of circular or rectangular section, or in small cotton sacks, filled and sewed by automatic machines.

"Grainer" salt, a coarser grained product than vacuum-pan salt, is made in elongated shallow pans, built of steel or concrete and heated by steam coils. The salt formed is removed by mechanical scrapers, moving

back and forth along the bottoms of the pans. They work with low-pressure steam. The texture of the salt prepared by the slow evaporation in grainers is quite different from the granular vacuum-pan product and from rock salt. Grainer salt is flaky or has thin flat grains. After leaving the grainers, it may be centrifuged until it contains about 6 per cent moisture, then it is artificially dried in kilns. It is desirable to have it rather coarse, grading from 10 to 40 mesh. The less it is handled, the better, for handling reduces the proportion of coarse salt.

In making butter, flake grainer salt of 10 to 20-mesh size is commonly used; and in cheesemaking 20 to 40 or even 60-mesh size is often used. The finer grades do not always find a ready market and are disposed of as advantageously as possible. Some grainer salt may be shipped unsized.

Solar salt from sea water is made in certain parts of California, notably along the shores of San Francisco, Monterey and San Diego Bays and at Long Beach. It is also made at Saltair along the shore of Great Salt Lake, Utah. More than 95 per cent of the California output is produced originally in this manner. Intake ponds are flooded from the bays at certain intervals at high tide. From these the salt water is pumped into secondary ponds or basins and gradually transferred from pond to pond as the brine strength increases. The concentrated brine or the crude solar salt is finally treated according to the kind of salt required, or may be sold in the original form for certain purposes. Solar evaporated salt is much more extensively made in southern Europe, in Asia, Africa, Oceania and South America than in the United States.

The preparation of rock salt for market consists of crushing and screening the mined product. For details see references 6, 12 and 22. At Retsof, N. Y., lump salt is produced 6 in. or larger and is shipped either loose or in bags. Other sizes are No. 2, No. 1, Coarse C, and Fine C, sized as follows:

	THROUGH MESH	OVER MESH
No. 2.....	$\frac{3}{8}$	$\frac{1}{4}$
No. 1.....	$\frac{1}{4}$	$\frac{5}{16}$
CC.....	$\frac{5}{16}$	8
FC.....	8	

From the storage bins for these sizes, the salt is delivered to the measuring chutes wherein are measured the correct quantity of salt for the bags ranging from 56 to 200 lb. per bag. When a bag is filled, it is sewed by an electric sewing machine. The bags are piled into box cars by hand.

In Louisiana,<sup>22</sup> "the rock salt is prepared in two general classes or groups of grades: 'coarse,' from the coarsest grade of rock salt, about  $\frac{3}{4}$  in. to 18 mesh; and 'fine,' from 18 mesh to the finest marketable size, about 50 mesh. There are no recognized standards of size, though the



same designation of size or grade by different producers represents a very similar product. The grades and corresponding limiting screen sizes are approximately as follows:

“Coarse Grades:

No. 4, through  $\frac{3}{4}$  in. on  $2\frac{1}{2}$  mesh.  
 No. 3, intermediate between No. 2 and No. 4.  
 No. 2, through  $\frac{5}{8}$  in. on  $\frac{3}{8}$  in.  
 No. 1, through  $2\frac{1}{2}$  mesh on 4 mesh.  
*A* grade, through 4 mesh on 6 mesh.  
*C* grade, through 6 mesh on 12 mesh.  
 Granulated, through 10 mesh on 18 mesh.

Fine Grades:

Through 18 mesh on 24 mesh.  
 Through 18 mesh on 35 mesh.  
 Table, through 24 mesh on 35 mesh.

“Another size sometimes made from the first screening of the rock salt, called ‘stock,’ is through 12 mesh on 30 mesh. The ‘granulated’ is usually a reground, kiln-dried product.

“The ‘granulated’ grade here placed in the coarse grades is sometimes grouped with the ‘fine’ grades. Another fine grade is sometimes produced, through 35 on 50 mesh, but it is not considered a standard size.”<sup>22</sup>

During the last three decades or so, pressed blocks of salt have come into the market as a substitute for the large lumps of rock salt used in field and stable for salting cattle. They weigh about 50 to 60 lb. and are made in hydraulic presses under great pressure, sometimes as high as 20,000 lb. per sq. in. Fine grades of evaporated or rock salt may be used in their manufacture, but the salt should be dry. Imperfect blocks are sawed into smaller blocks or bricks. Blocks are made sometimes with a small admixture of sulphur, supposed by some to give them medicinal value. Some concerns produce a mixture known as “smoke salt,” for curing meat at home, which is said to contain sugar, seasoning, a small quantity of pyroligneous acid and probably niter.

#### MARKETING

The mode of occurrence and geography of distribution have an important effect on the economics of marketing, speaking now of our own country. For a low-priced commodity like salt, sales territory is limited largely by freight rates, therefore prices must be kept at the lowest point. There is an area around each producing center outside of which freight rates are so high that competition from other centers of production becomes at first a limiting and then a deciding factor. This, of course, is why the production of salt has become so widespread in the United States and why certain of the larger manufacturers have plants in more than one district. Each area of occurrence has its own group of plants or mines, where there is a market. This condition has led to strenuous competition. This in turn has resulted in keeping prices down and has led to the elimination of the smaller and less efficient plants or their absorption by the larger and more up-to-date operators.

The primary production of salt is carried on in about 15 states, but the recognized producing areas are given by the U. S. Tariff Commission as 10 in number, which can be fairly closely circumscribed, as follows:

1. Western central New York.
2. Northern and central Ohio.
3. Southern Ohio and near-by parts of West Virginia.
4. Michigan (*a*) Detroit and vicinity, (*b*) Manistee and Ludington.
5. Central and southern Kansas and near-by parts of Oklahoma.
6. Louisiana in the vicinity of Weeks, Avery and Jefferson Islands on the Coastal Waterway.
7. Eastern Texas and Winnfield, La.
8. Utah, at Salt Lake and Redmond.
9. California: San Francisco Bay area.
10. California: Los Angeles, San Diego and Saltus.

The distribution of salt in dry form by the primary producers can be divided somewhat arbitrarily into three major classes, which roughly reflect grade, kind of shipping unit, and use. These classes, which do not correspond, as it happens, to those in the tariff act, are defined as follows:

1. "Industrial" salt includes all salt, regardless of quality, sold primarily for industrial uses and shipped in bulk, buyers' sacks, or producers' sacks, over 50 lb. in size.

2. "Domestic" salt includes all salt sold primarily for domestic and household consumption, and shipped in units such as cartons and packages in sizes up to and including 50-lb. bags and sacks, together with all barrels. Barrels are included here because "packets" or small bags are generally packed this way and there is now understood to be little shipment of loose salt in barrels.

3. Pressed "blocks" in all sizes, but usually 50 to 60 lb. Such blocks are used mostly for cattle licks.

The following notes taken from the U. S. Tariff Commission cover its classification used in the preparation of certain of its tables:<sup>20</sup>

While both systems of classification are functions of use and marketing unit, no system of classification applied to salt is entirely satisfactory and an exact alignment of the two systems is impossible, owing to the inherent irregularities and overlapping, as between classes. For example, a certain quantity of "industrial" salt constantly moves into channels largely supplied by "domestic" grades and sizes and vice versa. "Buyers sacks," a marketing unit peculiar to the native "industrial" class of salt, fills the requirements of industries such as dairy product manufacture, needing a steady supply of salt of definite specification and purity at a minimum cost. It has no significant counterpart in the international trade of the United States in salt and it is therefore regarded as a bulk form of salt for comparative purposes with imports.

On the other hand a very considerable proportion of salt imported in 200-pound bags goes into the tanning of fancy calf skins—a strictly "industrial" classification—comparable to native salt in producer's sacks. For such reasons the combination of producer's sacks and "domestic" salt is regarded as most nearly comparable statistically to imported salt packed in any form.

Analysis of the marketed salt in the United States shows that the output of dry salt has remained steady throughout the depression; and for a recent five-year period, 1929-1933, 77 per cent of the total output of dry salt was marketed as "industrial" salt, 19 per cent as "domestic" salt and 4 per cent as blocks. The principal producing areas, in order of total tonnage of dry salt produced were New York, Michigan, Kansas and southern Louisiana. The largest salt market in the United States is that for "industrial," salt in the northeastern and northeast central section. Of all salt of domestic origin entering these sections, 80.7 per cent and 76.8 per cent, respectively, was "industrial" salt. Combined, these two sections took about 60 per cent of all native "industrial" salt marketed in the United States. These two sections are likewise the principal markets for "domestic" salt, while the northeast central and western central sections comprise the principal market for blocks.

The significance of the distribution as between rock and evaporated salt lies in the fact that rock salt has an average manufactured value of about half that of evaporated salt. Consequently, rock salt is found to predominate in the "industrial" class of salt and evaporated salt in the "domestic" class. Also, from the standpoint of cost of marketing, the percentage of "industrial" salt moving in bulk is important, averaging 51.6 per cent of all salt, and when combined with other bulk forms, such as buyers' sacks and blocks, amounting to 61.8 per cent of all dry salt during the recent five-year period referred to.

It cannot be said of the salt industry as a whole that there is a maximum or minimum demand depending on the season of the year. As a rule the industries that consume the bulk of the salt do so uniformly from month to month throughout the year, changing only with the more fundamental fluctuations in the world of business and finance.

The chief marketing points are the large industrial centers where those industries implied in the list of uses are located. The bulk, therefore, is used in the large cities of the country and the uses have a specific relation to the chief marketing centers—meaning the selling or consuming points. For example, large quantities of salt are consumed in the textile industry in the New England and Southern States; in the tanning centers of the New England, the Middle Atlantic and the North Central States, including Illinois, Michigan and Wisconsin, especially the Chicago and Milwaukee districts; also the Johnstown and Gloversville districts, New York. Large quantities are used in the dyeing industry in New England, northern New Jersey, in the Carolinas and Georgia, and in the soapmaking centers around New York City, Cincinnati and Milwaukee. In such coast cities as Gloucester and Boston, Mass., considerable tonnages are used in curing fish. It is evident that this list of locations could be expanded considerably.

#### TESTS AND SPECIFICATIONS

The Food and Drug Administration of the United States Department of Agriculture defines table and dairy salt as fine-grained crystalline salt, containing, on a water-free basis, not more than 1.4 per cent of calcium sulphate ( $\text{CaSO}_4$ ), not more than 0.5 per cent of calcium and magnesium chloride ( $\text{CaCl}_2$  and  $\text{MgCl}_2$ ) nor more than 0.1 per cent of matters insoluble in water.

"Pending further announcement, no exception will be taken by the Food and Drug Administration to table salt that meets the requirements of the standard except that it contains anhydrous calcium sulphate (anhydrite) in excess of 0.1 per cent, provided that the total calcium content does not exceed 1.4 per cent."

The State of New York has no specifications except that the salt be pure and handled under sanitary conditions. In general the state accepts the Federal Government definitions of foods and food products. This presumably holds for the different states in general.

#### USES

Some of the uses of salt necessarily have been touched on under previous headings. It is employed both in the solid form and in solution. Its largest use is in the chemical industry, using this term in its more restricted sense, where it serves directly or indirectly in the manufacture of a long list of sodium chemicals, among which the soda alkalies take the lead.

In abattoirs and packing houses, salt is used in pickling or salting hides and casings. On the farm, it is used in fertilizers, salting hay, in stock feeds, and as a weed killer.

The food-manufacturing industry consumes large quantities of salt in preparations for animal and human use; in bread, cake, crackers, pretzels, and prepared flours of various kinds; in canning; in the preservation of butter and cheese and the clearing of oleomargarine; in the manufacture of condiments, sauces, dressings, etc.; in the curing and preservation of fish, meat, olives and pickles. The salt that is used in natatoriums for bathing purposes and in making medicines, medicinal waters, bath salts, and toothpaste comes under the head of human consumption. In the home, its uses are so well known that they do not have to be enumerated. Here it is used on a smaller scale, but chiefly for the same general purposes as in the large food and packing industries; namely, to keep products from decaying and to give them a flavor. It is necessary in the animal economy.

Salt is consumed also in many industrial and metallurgical processes and as a refrigerant. For example, it is used in the clay and clay products industry; in the manufacture of dyes and in dyeing; the glass industry; iron and steel; in knitting mills, laundries, the lumber industry, paper and pulp manufacture, the rayon industry, rendering plants, in the manufacture of soap, textiles, in tanning and tobacco manufacture. In some of these industries, its main use may be as a regenerator in zeolite water softening.

As a refrigerant, salt is used in freezing and packing ice cream and also in refrigerator cars. On railroads and highways it is used to thaw ice and snow, as a weed exterminator and in other ways. It is also used

in the production of what is called eutectic brine.<sup>15</sup> Doubtless many other uses have been omitted but enough have been given to indicate the vast importance of this saline in everyday life.

The chemical industry uses the largest quantity of salt. It is computed that in 1933 there was consumed by this industry in the United States 51 per cent of the total consumption. By far the most important of these chemical industries is the alkali, which includes the manufacture of all the different densities of soda ash, bicarbonate of soda, all densities and forms of caustic soda, causticized ash of various tests, modified sodas for the laundry, lumber, tanning and textile trades, the sesquicarbonate and sal soda.

If the size of the alkali industry, with its ramifications in the basic industries and in everyday life are considered, some comprehension is obtained of the indirect importance of salt in the daily economy, an importance which it must be stated is but little appreciated. The value of the sodium chemicals produced directly or indirectly from salt greatly exceeds the value of the salt itself. In 1933, the latest year for which United States Government statistics are available, the value of these sodium salts exclusive of salt itself and the hydrochloric acid, metallic sodium and chlorine produced as such was close to \$100,000,000, while the salt marketed as such was valued at only \$22,318,000. The soda alkalies alone were valued at more than \$53,000,000. Some of these sodium salts besides the alkalies are the acetate, benzoate, bichromate and chromate, bromide, chlorate, citrate, cyanide, fluoride, hypochlorite, iodide, phosphate, including the monobasic, dibasic and tribasic salts, the silicate, silicofluoride, niter cake (sodium bisulphate), normal sodium

TABLE 7.—*Consumption of Salt in the Process Industries, 1933<sup>a</sup>*

Product	Quantity, Short Tons	Per Cent
Soda ash.....	2,585,000	67.2
Caustic soda.....	421,000	10.9
Electrochemicals....	175,000	4.6
Fine chemicals.....	10,000	0.3
Coal-tar products.....	300,000	7.8
Fertilizer.....	5,000	0.1
Leather.....	100,000	2.6
Soap.....	100,000	2.6
Glass and ceramics.....	50,000	1.3
Pump and paper.....	50,000	1.3
Textiles.....	40,000	1.0
Vegetable oils....	10,000	0.3
	3,846,000	100.0

<sup>a</sup> Compiled by Lee.<sup>12</sup>

sulphate (salt cake), including the refined anhydrous, Glauber's salt, thiosulphate, sulphide, sulphite, tartrate, and others. The metal sodium itself is also produced from salt, together with considerable tonnages of hydrochloric acid and chlorine, the latter alone valued at more than \$6,000,000 in 1933.

The way in which the most important substances derived from salt enter the industries and trade cannot be detailed here, because this would be outside our subject; in certain branches these uses are extensive.

An interesting table published early in 1935 (Table 7<sup>13</sup>) classifies and estimates the consumption of salt in the process industries in 1933. The total of the salt so used is about 51 per cent of the total output for that year.

### PRICES

Prices are nearly always quoted in carload lots f.o.b. producer's plant, and depend on cost of manufacture. This means that at a given point a producer must absorb the freight rate differential if he desires to obtain the business. Some foreign salt comes to Atlantic ports spasmodically. At such southern ports as Baltimore and Norfolk, freight on the all-rail haul from northern points would greatly exceed the cost of bringing salt from European ports as ballast. Under such conditions it is conceivable that salt from northern producers might even be sold at times at a loss to hold customers during the periods in which foreign salt is not available in order to keep up plant production. In the manufacture of salt, losses are considerably enhanced if plants are allowed to close down. From what has been said it is evident that the sum total of various quotations for delivered salt is so great that to give them all is impossible.

In the salt industry competition certainly may be said to be the life of trade. Quotations are based on the quantity of salt sold or contracted for, according to the period of time covered by the contract, the purity or character of the product, the size of grain or brand of the salt, or whether it is sold in bulk, barrels, bags, or the smaller packages for household purposes. A multitude of different private brands, trademarks and packages exists, and as in the other mineral industries, sales depend on selling ability.

The foreign salt reaching coast ports comes in as ballast with little or no freight charge. To make the quickest possible turnover, it is sold cheaply, often at the foreign or home-market value plus duty if any, plus clearing-house charges. When there is no duty—for example, when it is used in the food industry in accordance with the recent tariff provisions—it is so much the cheaper. Not being a steady supply, however, it has no permanent effect on the market.

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## CHAPTER XXXVIII

### SAND AND GRAVEL

By J. R. THOENEN,\* MEMBER A.I.M.E.

**S**AND and gravel are perhaps the most familiar minerals of everyday life and their names are in common use. An accurate definition of either, however, presents difficulties. Search of the technical literature uncovers a surprisingly wide variation among authorities,<sup>11</sup> but the consensus would seem to be that sand is the unconsolidated granular material, coarser than 200 mesh and finer than  $\frac{1}{4}$  in., resulting from the natural disintegration of rocks; and that gravel is the similar unconsolidated granular material, coarser than  $\frac{1}{4}$  in. and finer than  $3\frac{1}{2}$  in., resulting from the same causes.

Both sand and gravel may be composed of materials in which a single mineral predominates or of a mixture of many minerals. Residual sands and gravels and glacial drift often contain appreciable quantities of the relatively softer materials, such as limestone and feldspar.<sup>121</sup> By far the larger proportion of gravels, however, is composed of the harder or more resistant minerals, such as quartz and various silicates, while sands as a rule are predominantly silica represented by quartz particles.

Residual sand and gravel particles are sharp-edged or angular, whereas transported gravels and sands consist of subangular to well rounded or water-worn particles.<sup>125</sup> Glacial gravels may be either angular or rounded, and individual particles may show abrasion scratches. Color varies from that of pure white quartz through various shades to the black of magnetite sands. In general, color is due to the mineral ingredients of the sand or gravel or to a thin scale covering the particles. Residual sands and gravels vary in hardness with the individual particle. Transported gravels, on the other hand, are largely composed of dense, wear-resistant particles approaching quartz in average hardness. Loose, dry sand ranges in weight from 90 to 110 lb. per cu. ft. and loose, dry gravel from 90 to 107 lb. per cu. ft. The weight sometimes exceeds maximum figures, and with moist material it may be below the minimum.

In some localities, where natural sand is scarce or of poor quality, artificial sands have been introduced with some success, sometimes in

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<sup>11</sup> References are at the end of the chapter.



direct competition with natural sands. Artificial sand may be the otherwise wasted fine product of crushed-stone plants properly processed, or it may be stone, crushed and ground purposely for sand production. In either case, the fine material is carefully screened into various particle sizes and these sizes are then recombined, so as to approximate closely the size gradation of natural sand.<sup>117</sup>

Roughly, commercial sand and gravel deposits may be classified in four groups with reference to their method of formation, as follows: (1) residual; (2) fluvial; (3) marine and lake; (4) glacial.

Residual deposits are commonly unstratified, heterogeneous mixtures of boulders, pebbles, sand and clay. They may contain much soft or inferior material, which through prolonged weathering has become porous and weak in structure. The particles may be angular or rounded, with rough or granular surfaces. Residual deposits commonly form a rock mantle over the parent formation. Ordinarily they are so intermixed with clay as to have little commercial importance.

Sand and gravel picked up and transported by stream action form fluvial deposits. Such deposits may be imperfectly stratified, and frequently they show rough size gradation. Coarse sand and gravel may be interspersed with lenses of fine sand or clay. Deposits have their long axis parallel to the direction of the stream flow. The beds usually vary greatly in thickness and may be complex in composition. As a rule, particles are poorly sorted and may be angular or rounded, depending on hardness and distance transported. When rounded, the surfaces are typically smooth.

Fluvial deposits are found in a variety of structural forms and usually overlie an eroded rock floor, but may occur above finer materials. Present rivers are constantly constructing, tearing down, and reworking bars within their banks. During periods of high water rivers leave their banks and extend over wide flood plains, leaving considerable material behind as they recede. At their mouths they deposit their loads in delta formations. Swift mountain streams emerging from narrow canyons or gorges onto broad, flat plains deposit their material in alluvial fans or cones. Old streams in wide valleys leave terraces behind as they cut their way down to lower levels. These are all typical fluvial deposits.

Marine and lake deposits frequently consist of well sorted materials with coarse and fine particles segregated. The various segregations usually have their long axes parallel to the shore line. Individual particles of marine sands range from angular to well rounded, while pebbles are usually well rounded and smooth. Marine deposits consist ordinarily of hard, tough materials received from streams and reworked by wave, tidal and marine-current action. Occasionally pebbles of limestone of organic origin, such as corals, may be present. Sand and gravel collected in lake beds present structures similar to marine deposits,

except that the materials are not usually so well sorted and cleaned. They are also less apt to be well rounded.

Glacial deposits are confined to areas that have been covered by the great ice sheets of the past. The ice originating about Hudson Bay, where igneous rocks predominate, brought down huge quantities of boulders, forming sand, and gravel of igneous origin now scattered over much of the northern United States and southern Canada. The ice also scoured out valleys in sedimentary rocks en route and carried along vast quantities of this material. Therefore glacial materials are composed of a mixture of hard and soft rocks. There are few deposits of sand and gravel that do not present evidence of more than one formational agency.

#### POLITICAL AND COMMERCIAL CONTROL

The sand and gravel industry is essentially a domestic industry. During 1932 to 1934, inclusive, domestic production was 344 million tons and imports exceeded exports for the same period by only 250,000 tons, or 0.07 per cent.

A tabulation for 1927\* of production by commercial companies showed that 50 per cent produced less than 25,000 tons each and less than 5 per cent accounted for 50 per cent of the total commercial production. A similar table† giving production by individual plants for 1931 gave 59 per cent producing less than 25,000 tons and 6 per cent accounting for 50 per cent of the total commercial production. These tables indicate a slight trend toward smaller individual plants. In further illustration of this apparent trend, it is observed that from Bureau of Mines reports the average annual production at commercial sand and gravel plants in 1927 was 104,500 tons. In 1931, this had dropped to

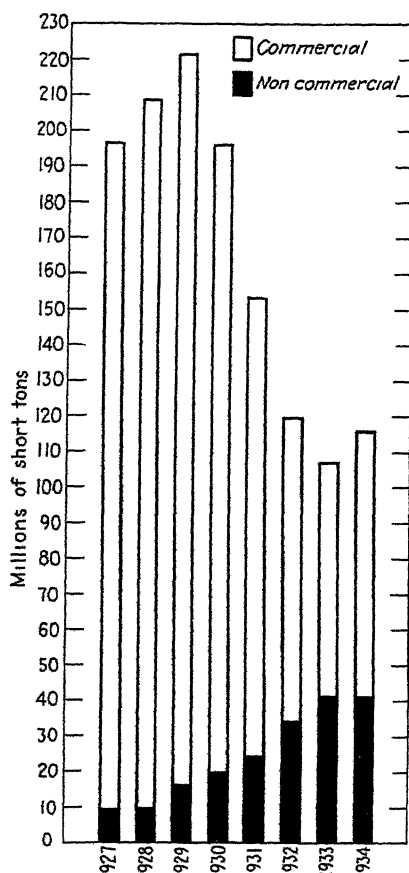


FIG. 1.—SAND AND GRAVEL PRODUCTION.

\* Mineral Resources, U. S. Bur. Mines, 1927, 163.

† H. H. Hughes and E. R. Phillips: Sand and Gravel, U. S. Bur. Mines Minerals Year-book (1932-33) 609.

59,300 tons and in 1934 it had decreased still further to 39,100 tons. This is partly explained by the increasing number of portable or semi-portable plants, which can be set up on local deposits for the purpose of avoiding long and expensive freight hauls.

During this period, however, the nation's consumption of sand and gravel had declined by 1933 to 50 per cent of 1927 production, but showed a 10 per cent increase in 1934. The apparent trend toward smaller plants may be accounted for somewhat by a smaller production from existing plants due to decreased demand. Both these tabulations

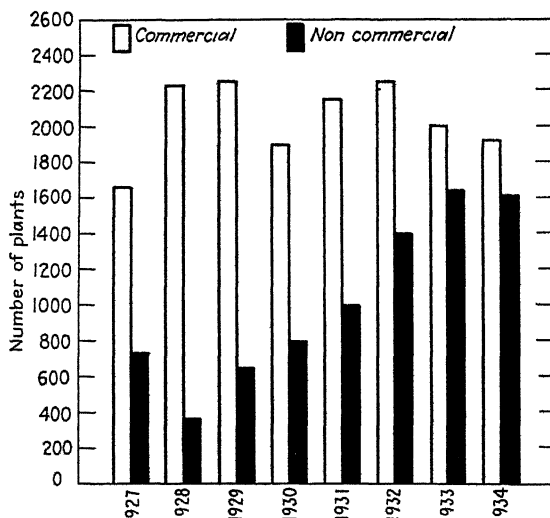


FIG. 2.—NUMBER OF PLANTS PRODUCING SAND AND GRAVEL.

refer to commercial production only. Hughes\* shows diagrammatically the growth of noncommercial production from 1928 to 1934.

Assuming that all noncommercial production following 1927 was from plants averaging 25,000 tons annually, the author has prepared Fig. 1 to illustrate the growth of noncommercial production and Fig. 2 the growth in number of plants.

#### PROSPECTING AND EXPLORATION

Prospecting for sand and gravel is usually done by one of two general methods: The direct method, wherein the prospector sets out to find a visible deposit; and the indirect method, wherein he relies upon topographic or geologic knowledge to guide him to likely localities.

In the direct method, the prospector must systematically examine the whole area for outcrops above the soil or water level, for visible deposits in clear shallow water, in railroad or highway cuts, or any known to exist

\* H. H. Hughes and M. Allen: U. S. Bur. Mines Minerals Yearbook (1935) 939-948.

from data collected in the digging of cellars, wells or post holes. A large percentage of the results will be negative.

The indirect method presupposes a knowledge of the structural geology of the region under investigation. This method is primarily one of elimination, in which the prospector avoids all the area that has unfavorable topographic or geologic structure. His first procedure is to collect all available data from the published reports of Federal and State geological surveys.<sup>31</sup> After finding favorable structures a knowledge of the genesis of the formation may give valuable information as to the character and value of the deposit itself. Table 1 shows the principal guides to surficial examination for sand and gravel deposits.

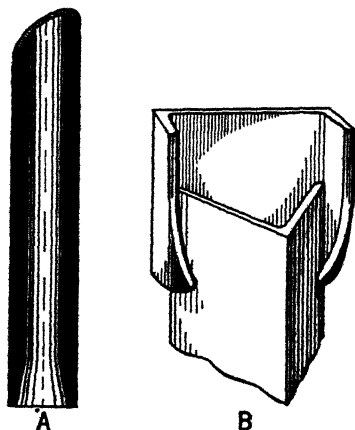


FIG. 3.—A, SECTION OF TEST-PIPE BIT; B, ANGLE AND CHANNEL.

So far as the writer is aware, geophysical methods have not yet been used by operators in prospecting for gravel deposits. During the summer of 1931, the Illinois State Geological Survey conducted some experiments with the relative resistivity method in locating gravel.\*

Following discovery of a promising deposit, the prospector must demonstrate that his discovery has a commercial value. This involves a careful examination of the material below the soil mantle. The size or extent of the deposit is important but of equal or greater importance is a careful sampling and testing of the material to ascertain accurately its natural size gradation and physical characteristics.<sup>33</sup>

The object of exploration is to determine the size, shape and value of a deposit.<sup>127</sup> It requires actual physical examination of the material over the whole area. This means opening holes or pits at intervals from which samples may be collected that will represent as nearly as possible the deposit as a whole. These samples must then be carefully tested and the results charted or otherwise recorded.†

\* A.I.M.E. *Tech.* Pub. 463.

† U. S. Bur. Mines *Bull.* 121, 259.

TABLE 1.—*Key to Sand and Gravel Deposits*

Formation	Sites of Deposition	Structure	Fine Material	Coarse Material	Associated Material	Relations to Underlying Material
Residual. . . .	Regions of disintegration. Tops of low rounded ridges between drainage basins and outside glacial areas.	No true bedding. Angular or spheroidal weathering. Heterogeneous mixture.	Disintegration sands and clays such as feldspathic clay from granite.	Well rounded (spheroidal weathering) to angular particles with rough or pitted surfaces. Flint nodules from limestones.	Associated with the parent rock because there has been no transportation	Boulders of weathering grade down into the unweathered rock. Differentially weathered deposits lie upon roughened surface of the parent rock.
Fluvial.....	Alluvial cones and piedmont plains. Terraces	Varied texture. Sand lenses common; often cross-bedded. Elongate parallel to stream flow. Material accumulated in discontinuous layers. Beds vary in thickness. Sorting may be poor. Matrix may be so abundant that pebbles do not touch one another. May be hundreds of feet thick in places.	Poorly sorted. Angular to rounded grains. More apt to be rounded.	All sizes up to several tons in weight, but mostly less than 6 inches in diameter. Subangular to well rounded. Smooth surfaces.	Sands and sandstones. Clay seams.	May lie upon eroded rock floor. Sometimes upon finer sediments
Marine and lake. . .	Stream channels Flood plains Deltas Nearly all high on beach. Hills between drainage basins in coastal plains	Pebbles usually touch one another. Well sorted. Possibly definite arrangement. May be pell-mell structure. Sand lenses elongate parallel to shore line. Rarely over 100 feet thick	Clean sands fairly well sorted. Angular to rounded.	Well rounded, smooth with dull polish	Sand and sandstone.	May rest unconformably on wave-scoured rock.
Glacial.....	Area originally covered by the ice.	No true bedding. Isolated nests and small beds of sand. Unstratified mass of miscellaneous unsorted rock.	Angular Decomposable minerals such as feldspar may be present. Rock flour containing sand grains may occur.	Angular or subangular striated surfaces. All sizes.	May be associated with water-washed materials.	If unconsolidated the underlying materials are apt to be disrupted and contorted. If rock, it may be striated and grooved.
Glaciofluvial.....	Valley trains; outwash plains; eskers.	Similar to fluvial alluvial cones. Stratification good, poor or absent. Structure often pell-mell.	Poorly sorted; angular and subangular predominate. Feldspar may be present.	Typically subangular. Large striated boulders.	Sand or sandstone.	Glacial abrasion on bedrock below. May rest unconformably on other deposits

If the soil is loose, the gravel below can often be outlined by means of an iron bar. By churning, the bar can be forced through the soil by hand. When it strikes gravel, a distinctly different metallic sound will be heard, and usually the gravel will be more difficult to penetrate than the soil. A length of  $\frac{3}{4}$ -in. pipe with a steel point welded to one end may be used for deeper holes. This method is applicable to the depths of 2 to 15 ft. The method is inconclusive in that it recovers no samples and is useless in thick or tight overburden. Some operators use ordinary steel pipe, 2 to  $2\frac{1}{2}$  in. in diameter. One end of the pipe is forged or filed to a sharp edge, with the bevel on the inside (Fig. 3A). A malleable-iron coupling or tee is screwed to the other end and the pipe is driven into the ground. Old diamond-drill rods can be used for this purpose with less friction. The pipe is pulled, held over a wooden or steel platform, and tapped with a light hammer. As the material falls out the pipe is moved in a straight line. In this way the material is strung out in a sequence corresponding to its position in the ground. In deposits containing many cobbles or boulders, pipe cannot be used successfully. Pipe testing, as a preliminary only, is valuable both for outlining a deposit and for collecting a rough qualitative sample.

Earth augers or post-hole diggers are often used to determine the thickness of overburden. One Texas operator uses a machine-driven post-hole digger, cutting a hole 16 in. in diameter.<sup>5</sup> The machine is operated by a gasoline tractor and digs from 1 to 5 ft. per minute in clay overburden. It is not successful in digging gravel or in ground containing boulders.

A recently patented method of obtaining subsoil samples is described in an article in the *Engineering News Record* (March 3, 1932). A 4 by 4 by  $\frac{5}{8}$ -in. angle is driven vertically into the ground at the desired point. A 7-in. (20-lb.) channel is then driven alongside the angle in such a position that the channel web spans the outstanding legs of the angle (Fig. 3B). The two shapes are then pulled and the enclosed material is removed intact. The lower end of the channel has the flanges turned in for a short distance so that it will overlap and follow the angle as it is driven. This method encounters the same difficulty as pipe driving when used in ground containing large boulders.

The churn drill is used by many operators with or without casing in exploring gravel deposits. By careful drilling and with the casing close to the bottom of the hole, fairly accurate information can be obtained. Churn-drill holes in gravel should be cleaned out and the casing driven down at not more than 2-ft. intervals.

For exploration purposes the gasoline-driven, caterpillar-traction churn drill is perhaps the most popular type of machine. It supplies information as to the depth of overburden and thickness and quality of gravel. It can be successfully used to depths of 50 ft. or more. The

churn drill is usually the best device for exploring below water level. More accurate samples can be obtained by driving ahead of the drill, and using the latter only to break up the enclosed material before bailing. In ground that can be penetrated by driving casing, the drill may be eliminated and a specially constructed flap-valve bailer used. The lower, or cutting, end is fitted with a three-pronged tempered cutting edge. This tool is churned up and down, breaking up the material inside the casing, and by means of the valve at the bottom the sample enters the bailer and is removed. This method has many advocates for sampling material below the water table. A small orange-peel bucket is also sometimes used inside the casing. The bucket is operated by hand with cable and tripod, in the same way as the bailer. The casing must be driven ahead of the bucket. As is true of all methods involving casing, it is not successful where boulders are present.

The preceding methods are recommended for outlining the boundary of a deposit and for obtaining preliminary rough samples. None of these methods is recommended for accurate testing for gradation or quality. The only reliable and accurate method of exploring land deposits of sand and gravel is by means of test pits. They are more expensive to dig than post holes or churn-drill holes, but their accessibility for obtaining accurate information justifies the additional cost.

Sampling a deposit of sand and gravel is one of the most important functions of exploration.\* All too often a small amount of the dump from each test pit is taken from the most convenient parts of the pile and considered as representative of that pile. These portions are thrown together and subjected to careful and exacting screen analyses. The result is then considered as a screen analysis of the whole deposit. The author wishes to emphasize this error particularly, for he has found it to be very common in practice. The actual result of such a test is merely a screen analysis of the material used in the test.

Often operators are surprised and disgusted at the difference in the analyses of material submitted to two or more commercial testing laboratories. The testing laboratory can do nothing more than analyze the material submitted to it.<sup>6</sup> If the samples themselves are unlike, the results from different laboratories are bound to differ. Therefore, in submitting two samples of the same material to different analysts, great care must be taken to see that both truly represent that material. It is axiomatic that an analysis can be no more accurate than the sample. The care and accuracy of test methods too often far exceed that used in sampling.

The correct sampling of a lot of ore (material) is the process of obtaining from it a smaller quantity which contains unchanged percentages of all the constituents of that lot. As

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\* See U. S. Bur. Mines *Bull.* 121 and *Bull.* 259.

there are limits to the accuracy of weighing and determining the constituents *it is necessary only that the error in sampling be smaller than the error in testing.*\*

The first step in obtaining a representative sample of a deposit is the systematic placing of drill holes and test pits over the entire area. The next step, equal in importance, is the method of taking the sample itself. In sampling test pits the object is to obtain a continuous sample from top to bottom. If the walls of the pit do not require timbering, collection of such a sample is simple. One sampler holds a box against the wall while another, with pick or shovel, cuts a vertical channel in the wall 6 in. to 1 ft. wide and 2 to 6 in. deep. Collection of material in this manner should be stopped at regular intervals and a new box used. The sample taken should be carefully labeled with the number of the hole and the depths from and to which the material was removed. Where the gravel contains thin sand or clay beds, the boxes should be changed at each change in material. Where the deposit is uniform, 5-ft. intervals are sufficient. The sampler should keep a record in his notebook of any unusual occurrences in the deposit, such as single boulders or nests of boulders, water seams, changes in bedding, etc. His notes should also show from which wall of the pit the sample was taken. It is often advisable to take a similar sample from the opposite wall as a check.

Where the walls of a pit will not stand without timber, the sampling must be done as the pit is sunk. It is seldom that timber must be kept within 4 ft. of the bottom. In such cases, the wall channel samples are taken just prior to the placing of timber. The procedure is the same, except that each 5 ft. is sampled separately instead of the whole depth being sampled at one time. Deposits in loose, dry sand or quicksand are by nature more homogeneous in character, and vertical accuracy is not so essential. In such deposits it is often sufficient to take a channel sample across the floor of the pit at each vertical foot in depth.

When earth augers are used the material removed should be placed in line on the surface. It is then bagged in the predetermined vertical intervals, tagged, and recorded in the sampler's notes.

Sampling of material obtained by bailer or churn drill, or wherever water is used or encountered in the hole, presents other difficulties. Accurate samples are obtainable by churn drill or bailer only where the casing can be driven ahead of the drill bit or sand pump. Accurate sampling requires that for each vertical interval cut by the drill all material must be saved, whether coarse or fine. This means that the material in suspension must be saved with the coarse material. All material, including the water, as removed from the hole, should be placed in a box from which no water is allowed to escape before the fines settle out.

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\* R. Peele: *Mining Engineers Handbook*, 1730. New York, 1918. John Wiley and Sons.



When completely settled the clear water is poured off and the material in the box is bagged and labeled for hole number and depth as before.

When using the small clamshell bucket within casing, the coarse material should be removed and placed in a continuous pile or ridge. When the bucket has dug to the bottom of the pipe, the water and suspended material should be removed by a bailer and allowed to settle. This fine material is then added to the coarse material and thoroughly mixed before the sample is bagged.

#### DEVELOPMENT

Development is defined as the process of preparing a deposit for exploitation with the object of recovering its contents for sale or use. The decision as to the method of operation and type of equipment to employ is governed largely by the information obtained by prospecting and exploration. It will also depend upon a number of other factors totally outside the scope of the prospector's field, such as the production capacity desired, the market to be served, and the capital available. Totally aside from the technical problems presented in sand and gravel recovery but of equal or possibly even greater importance are the many economic problems involved. These may condemn a property that offers ideal operating conditions from a purely technical point of view.<sup>10</sup>

The physical characteristics of the deposit present the first problems in devising a development plan. A deposit elevated above the surrounding land surface requires a plan of operation different from that for one below the surface. Alternative equipment affords a choice of several methods of developing sand and gravel from two deposits, even though they may be of the same general type.

The size and shape of the deposit will have a direct effect upon the development plan. A small, irregularly shaped deposit does not lend itself to large-scale development. Another deposit, small in volume, may be so self-contained and regular in shape as to warrant large-scale operation. Long, narrow deposits may be opened transversely to best advantage on one site while at another they must be developed parallel with the long axis.

The proximity of the permanent ground-water level to the surface will affect the method of development of subsurface pits. Sand and gravel occurring in areas in which the ground water approaches the land surface provide a wide choice of excavating equipment. In thin deposits, otherwise suitable for operation by pump dredge, the water level may be too low to float the dredge.<sup>73</sup>

Development for dragline or scraper operation will be different from that for dredges. Different types of dredges require different development programs. For example, a pump dredge may excavate and deliver material through pipe lines to a plant on land, while a clamshell or ladder

dredge may deliver directly to barges to be towed to the land plant, or either type of dredge may be constructed to treat the gravel on the dredge itself, delivering finished material to be transferred to distribution yards or semifinished material for further treatment in the land plant.<sup>60</sup> Gravel under water may be developed for dragline, cableway or dredge operation.

The removal or utilization of overburden often presents formidable problems; on the other hand, stripping is sometimes inconsequential. Under-water deposits in general have no stripping problems. At times, however, floods may bring down a quantity of undesirable material and completely cover a commercial deposit.<sup>9,25</sup> Such conditions may require development behind jetties to divert river currents.<sup>62</sup> Few land deposits are devoid of overburden, although often such overburden does not present operating difficulties. If the overburden is thin or sandy, it may be feasible to disregard it entirely and send it to the treatment plant with the gravel. If thick, or not easily removed by washing, it must be stripped ahead of gravel excavation.

Stripping may be either periodic<sup>28</sup> or concurrent with production of gravel. In either case, it may be possible to use the same equipment for both stripping and gravel excavation.<sup>70</sup> Sometimes digging equipment is used for stripping during the winter or nonproductive seasons;<sup>45</sup> in other cases, the same equipment handles stripping when delays in plant or haulage equipment prevent mining. At other places, provision is made in the operating cycle for alternate stripping and production.<sup>91</sup> At still others, different or identical types of equipment are used concurrently, with one unit engaged in stripping and another in producing gravel. At some places, the overburden may have constant or periodic commercial value;<sup>27</sup> at others, local laws require the replacement of top soil after removal of the gravel. The development of a deposit stripped hydraulically will be entirely different from that of one stripped by mechanical equipment. Excessive thickness of an unsalable overburden may render a deposit of commercial gravel worthless with one method of stripping but workable with another.

Some deposits contain an excess of boulders requiring crushing and only a relatively small amount of sand.<sup>72</sup> Typical examples are some alluvial-fan deposits of the West Coast and the river deposits in the Pittsburgh area.<sup>27,67</sup> Other deposits, and these are far more prevalent, contain an excess of sand.

Still other deposits contain quantities of deleterious materials and require development methods different from those suitable for clean materials. As examples, a hillside deposit may contain such an excess of clay as to require preliminary washing by hydraulic excavation. An under-water deposit may contain such an amount of river debris as to prohibit the use of any other than a clamshell dredge.<sup>9</sup> In some river operations the material excavated in a season's digging may be completely replaced

by spring floods, thus permitting excavation from the same pit year after year from a stationary plant.<sup>37,38</sup> Development at such a deposit obviously is different from those requiring portable river equipment. A large deposit may contain commercial material segregated in certain portions or contain sand at one point and gravel at another.<sup>44</sup> Such a deposit, though large in areal extent, must be developed in small-scale separate units.<sup>36</sup>

Often sand and gravel are partly cemented by calcareous, siliceous, or metallic oxide materials,<sup>49</sup> extending throughout the entire thickness of the deposit or in only thin layers or beds. Such beds may be so tightly cemented as to prevent penetration by pump suction or, if dry, by dragline cableway or even a power shovel. Several instances are known where the gravel bank must be drilled and blasted ahead of even heavy shovel equipment,<sup>49</sup> and in one instance at least submarine blasting ahead of a ladder dredge was contemplated.

Thin deposits extending over wide lateral areas must be developed to provide easy access to an extended and rapidly changing working face. This requires light and mobile digging equipment and the haulage equipment or track layout must be suited to rapid and frequent changes.

Thick deposits may require working more than one bench by the same or different types of equipment. Even where a thick deposit is worked in one bench the methods of attack will differ greatly in different localities, either because of topographic or other physical conditions or because of personal preferences for different types of equipment. Bedded material in a thick deposit may require a different method of approach at one point than at another in the same deposit. Inclusion of lenses of waste material in the deposit may cause a change in method as excavation proceeds.

Some thin submarine deposits are recovered more economically by cableway or dragline than by dredge, even though there is abundant water to float a dredge.

Waste in a gravel pit comes from two sources—overburden and an excess of some one or more sizes of processed material. In the one case it originates at the site of the excavation; in the other, at the plant. In both cases, provision must be made for its disposal. Dumping space must be allocated where it will cause a minimum of future inconvenience and yet where it can be reached with a minimum of expense. A dump suitable for overburden may be entirely out of the question for plant waste, or vice versa. Periodic sale of either overburden or plant waste may require the dump to be placed to allow easy recovery of its contents. For the same reason, it may be inadvisable to mix overburden and plant waste.

There is probably no one feature having a more profound effect on development than that of the quantity of material that must be handled daily. Development of any deposit for small-scale production will have

little relation to that for large-scale operation. There will be a difference in the amount of stripping required, in the capacity and operation of digging equipment, in the haulage equipment, and in the treatment plant. Also, an entirely different method of excavation and treatment may be needed. As an example, the operator of a small wayside pit may find excavation by scrapers and tractors delivering to a log washer sufficient to recover, wash and eliminate an excess of fine sand. A large plant on the same site would require shovels, locomotives, cars or other conveying equipment and an elaborate washing, screening and possibly classification plant. The small operator has just as much need for proper exploration as the large operator. Development for the wayside pit requires only temporary provision for a relatively short period of operation and need not be concerned over the life of the deposit or equipment. Plant capacity, however, is usually determined by economic factors entirely aside from technical problems. When capacity is once determined, the plant equipment becomes a technical problem that must be solved by the development program.

The plant always must be made to fit the capital available. If it does not do this, the result is inevitable failure.

The method of delivery of the material to the plant is influenced by surface topography and depends largely on the type of excavating equipment used. Usually the first step in handling material at the plant is to elevate it to a considerable height, from which it may pass through the various processing steps by gravity. Occasionally, when material requires the elimination of excess fine sand or clay, a preliminary washing is given before this initial elevation, in order to conserve power by decreasing the amount of material elevated.

In dry surface deposits, one operator will depend upon auto-truck haulage from pit to plant, another will install locomotives and cars, a third may prefer a conveyer-belt system,<sup>103</sup> while a fourth may use hydraulic pumps and pipe lines. The same is true of wet or dry sub-surface pits, with the exception of a cableway excavator delivering directly to the plant.<sup>20</sup> Even in under-water deposits, the transfer of material may be effected in many ways, such as by cableway or scraper excavation directly to the plant,<sup>29</sup> by tugboat and barges, aerial tramways,<sup>50,59</sup> or by pumping through pipe lines.<sup>74</sup> Proper consideration of the haulage problem within the plant is of prime importance in any plan of development.

Material discharged from pipe lines may or may not require further fresh water for washing. Material delivered by trucks, cars, dragline or cableway invariably requires wash water. The provision of adequate sources of water and its delivery to the plant in the required quantity is as much a part of the development program as the preparation of the deposit itself. Some gravels require only washing to prepare them for market. Others require the elimination of foreign matter, which may

necessitate the use of special machinery, such as jigs.<sup>61,63</sup> Where sands are prepared for special markets, special machinery may be required, and provision should be made in the development program to accommodate this treatment.

It will be seen from the foregoing discussion that the development plan must consider a large number of operating details both as to their individual effect upon the sequence of operations and as to their combined effect in influencing the coordination of the several operating steps. Thus, while development in its restricted sense deals only with the preparation of the deposit for exploitation, it must in a broader sense encompass the causes, effects and results of a variety of operating methods and equipment and by preliminary planning incorporate the whole into a smoothly working sequence of operations designed to suit local conditions.

#### MINING METHODS

Two major factors govern the selection of a method to be used in mining a sand and gravel deposit—type of deposit and type of equipment used. The character of the deposit may limit the choice of equipment to a certain type of excavator and an equally restricted choice of haulage equipment. For example, a deposit covering a lake bed may limit excavation to a hydraulic dredge served by floating barges. Ordinarily, however, any deposit can be worked by different methods involving various types of excavating and haulage units. Two operators that have had experience with different types of equipment may operate the same type of deposit successfully by entirely different mining methods. Therefore no exact rule can be laid down definitely linking one type of deposit with any particular method.

The author has classified<sup>12</sup> sand and gravel deposits by types as: (1) *bank* deposit, one in which the elevation of the excavation floor is level with or above the surrounding land surface; (2) *pit* deposit, one lying wholly below the level of the surrounding land surface; (3) *marine* deposit, one forming the bed, bars or small islands between the banks of a natural body of water.

Pit deposits are subdivided into dry pits, in which the excavation floor is above the level of permanent ground water, and wet pits, in which the excavation floor is below the water level. Marine deposits are subdivided into river, lake and ocean, depending upon the kind of body of water.

Before mining methods applicable to sand and gravel recovery are discussed, surface mining methods will be defined and classified. Since the terms "mining method" and "mining system" are often used synonymously, it is first necessary to designate definitely what is meant by each. After considerable etymological research the author concludes that "system" is the major term and may include various "methods."

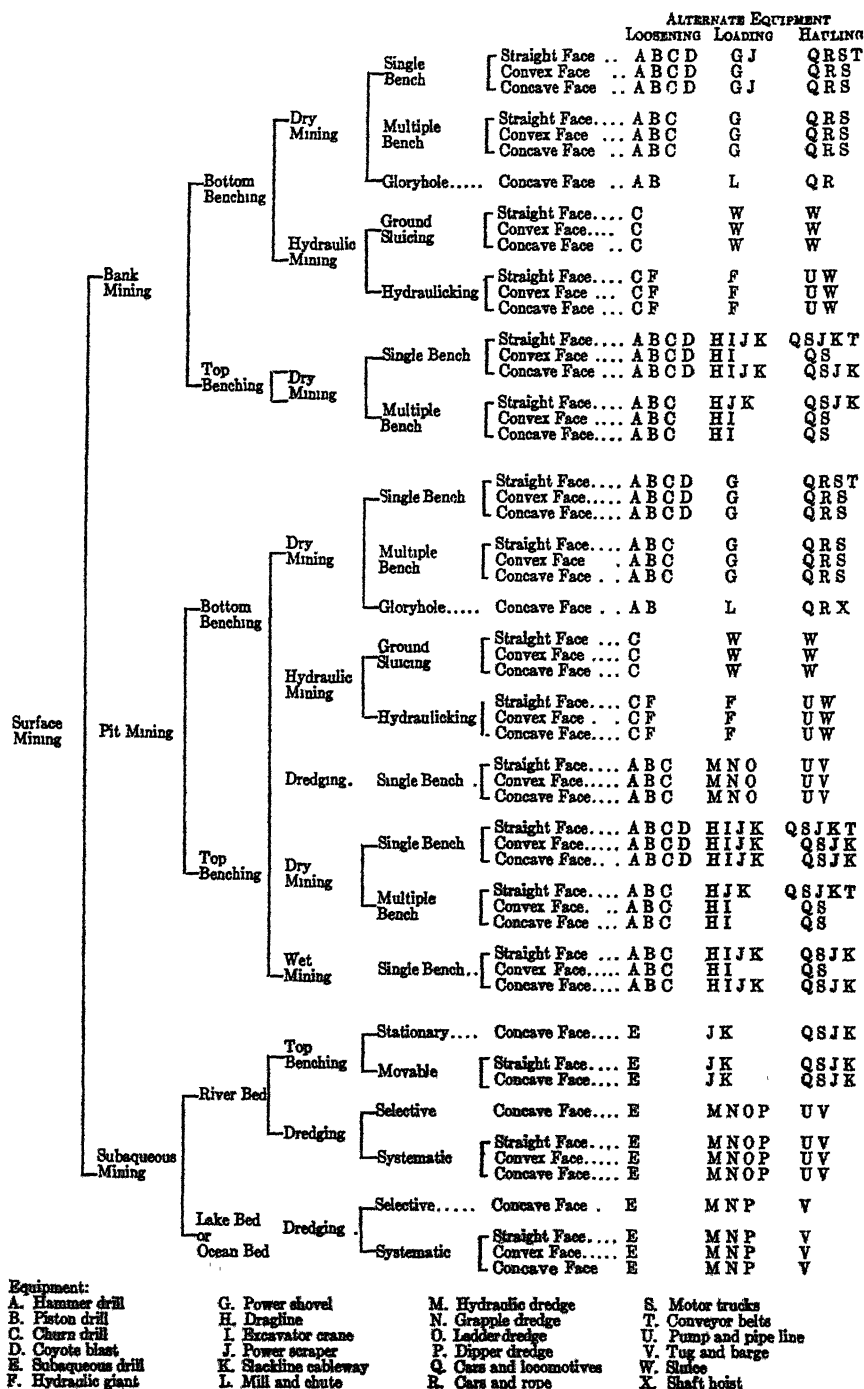


FIG. 4.—SURFACE MINING CLASSIFICATION.

Just as a country may have a system of taxation but uses various methods of collecting the tax, so each mine may have an orderly "mining system" that may embrace various "mining methods" that are more or less interdependent.

Fig. 4 shows the author's classification in chart form.<sup>18</sup> A study of the chart shows that the mining of sand and gravel deposits of the same type is not restricted to a single mining method nor to a system composed of identical methods. Variation may be small in some instances (two systems may be similar in general but differ in contributing methods) but may result in greater proportionate efficiencies. The problem of selecting the proper system for a given deposit is complicated further by the overburden covering most bank and pit deposits and some marine deposits.<sup>29</sup> If the overburden is thin enough to be negligible, it is removed in the process of mining the gravel;<sup>37, 46, 63, 89</sup> if it is thick enough to cause trouble it is usually removed separately. The stripping method may then parallel the mining methods, using the same<sup>51, 58, 86, 94</sup> or different equipment,<sup>36, 70, 76, 99</sup> or it may be entirely different from the mining method and require its own set of equipment.<sup>43, 54, 57, 94</sup> On the other hand, if different methods are necessary in stripping and mining, the same or duplicate equipment may sometimes be used for both.<sup>103</sup> However, removal of overburden or stripping is surface mining and is subject to the same classification of mining methods as is sand and gravel excavation.

Bank deposits are mined dry, except where hydraulic mining<sup>66</sup> is employed. Haulage from bank deposits is level or downgrade, with one exception, which will be discussed later. These two distinctive characteristics of bank deposits at once eliminate consideration of dredges and water-borne transportation, and permit the design of the haulage equipment for a minimum of power.

Bank deposits ordinarily are mined by bottom benching. The choice between single and multiple benches depends, first, upon the thickness of the deposit, and second, upon the type of excavator used. Single benches cut with power shovels usually do not exceed 50 ft. in height.<sup>114</sup> For deposits of greater thickness, multiple benches are recommended. Although shovels are sometimes used with high benches<sup>66</sup> precautions must be taken against dangerous caving, entailing loss of life or property damage. The power shovel has no means of protection against caving from a high bank. A precautionary method often employed is to mount a small hydraulic giant on or near the shovel,<sup>51</sup> the water from which is directed against the upper part of the bank to cut down the top ahead of excavation by the shovel at the bottom. Another method involves the use of a stream of water pumped to the top of the bank and allowed to trickle over the face and wash down the upper portion.<sup>66</sup>

Single, high benches permit the use of larger shovel units and require less frequent shovel movement, thereby increasing efficiency. On the

other hand, multiple benches necessitate the use of more—usually smaller but more mobile—units, permitting more selective mining from the face—an advantage in providing a more uniform feed to the washing plant. Banks higher than 50 ft. may be excavated safely and successfully as single bottom benches by power scrapers,<sup>73,92</sup> even when the bank consists of heavily compacted gravel, if the tail tower is placed on top of the bank and the head tower at the base. In such a setup, the scraper effectually breaks down the top of the bank and prevents dangerous caving. High banks operated as single bottom benches often permit excavation without previous stripping, even though the overburden may be fairly thick.

Where bank deposits must be stripped, the overburden is usually removed in a single bottom bench by a power shovel<sup>44</sup> or by hydraulicking, if water under pressure is available. Sometimes, however, the overburden is removed in a single top bench by dragline,<sup>86,111</sup> power scraper<sup>63</sup> or slackline cableway. The contour of the surface adjacent to bank deposits usually provides dumping space for overburden in ravines. Cars and locomotives or motor trucks serve the shovel and dragline, but often the power scraper or slackline cableway can dig and haul to waste in one operation. If no such space is available except by long haul, the operator should weigh carefully the cost of separate stripping with multiple benches against single bench without stripping, even though his washing expense may be increased by the latter combination. The power scraper excavates a bank as a bottom bench.<sup>63,92</sup> It can be made to cut a straight or even a convex face, if the inhaul tower is moved laterally with the tail tower.

Bank deposits may be worked with water by hydraulicking<sup>3,24,66,115</sup> to sluice or pump sump for haulage to the washing plant.<sup>14</sup> Ground-sluicing, commonly used in placer mining, is not used in mining sand and gravel. Glory-hole methods,<sup>116</sup> common to metal mining and quarrying,<sup>95</sup> seldom have been applied to sand and gravel deposits.

Operating a bank deposit as a single bottom bench has the advantage of concentrating all haulage operations on one operating level, whereas if multiple benches are used the haulage method must be designed to collect loaded equipment from each bench and deliver it to a main line over which it is lowered by proper grades, often necessitating long, curved tracks or switchbacks.<sup>114</sup> The power shovel may be served by cars hauled by locomotives<sup>75,108</sup> or by motor trucks.<sup>44,96</sup> If the face of the bench is kept straight field belt conveyers may be used,<sup>66,100</sup> but they must be of special design for curved faces.<sup>80,84</sup> The power scraper ordinarily does its own hauling, but may discharge over a ramp or hopper to any type of haulage, including cars, trucks, conveyer belt,<sup>63,92</sup> or even an aerial tram.<sup>50</sup> The power scraper or slackline cableway usually employs portable towers and loads some other kind of haulage—car, truck or conveyer belt.



Where banks are mined by hydraulicking, the giants wash the excavated gravel either to sluices,<sup>109,112,113</sup> which carry it to the washing plant, or to sumps cut in the floor, from which dredge pumps pick it up and transport it through pipe lines to the plant.<sup>117</sup>

Hydraulic mining and transportation have the advantage of washing the gravel before it reaches the plant. This is of major importance when the gravel deposit contains clay. On the other hand, water transportation through sluice or pipe line is difficult if the deposit contains more than 10 per cent of boulders. These must be removed either at the face or some other point between the face and entrance to the sluice or sump. Usually a crawler crane is installed for this purpose.

Sluices are suitable for haulage from a deposit elevated above that of the washing plant. Dredge pumps and pipe lines, on the other hand, can transport the material over level ground or elevate it directly to the top of the plant.<sup>99</sup> Both hydraulic mining and hydraulic transportation require large quantities of water, preferably from a source above the gravel deposit. Water from a higher elevation, however, is seldom available for bank deposits and must therefore be pumped, which adds to the expense.<sup>14</sup>

Excavation of bank deposits by top benching with single or multiple benches must be conducted without the aid of water. Top-benching bank deposits is not ordinary practice; it is applicable only under special local conditions, which make it analogous to pit mining by the same method.

Power shovels and hydraulic giants are unsuited for top benching because they have no means of elevating material. The equipment most commonly used for top benching includes the dragline, power scraper and slackline cableway. Stripping and mining over the same area in a bank deposit by top benching can be done from the same setting of the head towers of either the power scraper or the slackline cableway. If both operations are performed by the same unit the overburden must be loaded into haulage equipment for disposal, but if separate units are used, the stripper usually can be located so that it can excavate and haul the overburden direct to the waste dump. Where one unit does both stripping and mining the tail towers must be set for each operation. Using the same mining method but excavating by dragline, a single unit can both strip and mine from the same position by alternating from one operation to the other. Moreover, the dragline can load overburden to haulage equipment or cast it to the excavated area behind the gravel bank. This process carries stripping concurrently with mining and constitutes the most common application of the use of multiple benches to top-benching a bank deposit.<sup>13,14</sup>

The dragline may be served by cars and locomotives, motor trucks, or field belt conveyers if the bench faces are straight. The power scraper

and slackline cableway usually serve also as haulage units, but if mobility in both head and tail towers is required haulage units must be introduced between scraper or slackline and plant. In that method of multiple top benching in which a single dragline strips overburden and mines gravel concurrently, the method of haulage varies. The overburden and gravel may be loaded into different cars in the same train, separate trains on the same track, separate trains on different tracks or separate trucks. Overburden may be hauled by trucks and gravel by cars or vice versa, or the gravel may be delivered to hoppers feeding a field belt conveyer. The overburden may also be carried to the excavated area without requiring haulage equipment.

Pit deposits are subdivided into dry and wet pits, according to the presence or absence of subsurface or ground water. Virtually all dry pits are mined by methods similar to those used in bank mining. Methods used in wet pits differ materially from bank mining, although similar equipment may be used. In pit mining, whether dry or wet, the material must be elevated to a point above its natural position. The possible presence of ground water and the necessity for elevating the material are the chief differences between pit and bank mining. Pit deposits are mined by bottom benching with power shovels,<sup>64</sup> power scrapers,<sup>50</sup> or hydraulic giants; by top benching with cranes,<sup>83</sup> power scrapers<sup>39</sup> or slackline cableways;<sup>81</sup> or by dredging.<sup>87</sup> The last named, however, may be similar to bottom benching, depending upon the relation between the elevation of the water surface and that of the top of the bench or ground surface.

In mining dry-pit deposits by the bottom-bench method, the excavator in most common use is the power shovel. Either single or multiple benches may be cut by the shovel, depending upon the thickness of the deposit. With thin overburden over a 50-ft. deposit, stripping is often unnecessary.<sup>46,56,90</sup> Thick overburden may be removed as a single bottom bench followed by a similar bench in the gravel—a multiple bottom-bench method.<sup>51,103</sup> In the same type of deposit, the overburden may be removed by top benching, although the bottom-bench method is retained for mining gravel.<sup>54</sup> Pit deposits can be mined by hydraulicking in the same manner as bank deposits. Preliminary stripping may be done by bottom benching with shovel or hydraulic giant or by top benching with other equipment.

Top benching with draglines is common and economical. The dragline can strip overburden from a small area and cast it beyond the toe of the gravel bench in the excavated pit and then without changing position mine the gravel from the stripped area.<sup>85,87,99,110</sup>

The power scraper is frequently used to strip and mine pit deposits,<sup>76</sup> but both operations cannot proceed concurrently, as with the dragline, without contamination of the gravel with overburden. The usual pro-

cedure with power-scraper operation is to set up the unit and strip a predetermined area. From the same position, the gravel is then dug from the stripped area. Thus the actual operation is alternate stripping and mining from the same setup, but all the overburden must be removed from a given area first and the gravel later. By reversing the direction of haul the power scraper may be used to strip gravel and dump it around the tail tower and later dig and haul the underlying gravel to the head tower. By this method the bucket must be returned mechanically, and gravity return cannot be utilized.

The slackline-cableway excavator mines a dry-pit deposit by top benching in much the same way as the drag scraper,<sup>43,67,81</sup> except that the direction of haul cannot be reversed because the slackline depends upon gravity to return the empty bucket. The slackline has an advantage over the scraper in that the bucket is raised above the surface on the inhaul and can be used to strip one portion of the deposit and dump the overburden in the excavated area without contaminating the gravel face. The slackline, by carrying the loaded bucket high in the air, can often avoid obstructions in the line of travel that would have to be removed for power-scraper operation.

The type of haulage selected may determine the choice between bottom or top-benching a dry pit. For example, if top benching by dragline is selected, the haulage route may be of minimum length—from the dragline to the treatment plant. Excavation by bottom benching with a power shovel from the same point in the bench might require a long, circuitous route to provide the grades that would enable the haulage unit to ascend from the bottom to the top of the pit, plus possible additional surface haul around the perimeter of the pit.

With equal length of haul for both systems the type of haulage used will still be an important factor in the selection of the mining method. For example, a power scraper or slackline cableway may excavate material from a certain point in a pit and haul it directly to the plant on the surface over a distance equal to that required by a truck or locomotive serving a shovel at the same point. The power scraper excavates, hauls and elevates with one movement, whereas the shovel excavates only and the truck or locomotive must haul and elevate. Thus, the scraper or slackline requires no initial investment for service haulage equipment, or roadway grading and maintenance, and capital charges may be reduced considerably, with corresponding reduction in operating costs.<sup>103</sup>

The common methods of haulage from bottom benching by power shovels utilize cars and locomotives<sup>46,56,90</sup> or motor trucks.<sup>65</sup> As the maximum grade suitable for either type is fixed, the length of haul increases as the pit depth increases. The maximum grade for locomotives is less than for trucks; hence, for the same pit depth, the haulage distance for a locomotive is greater. Obviously, added length of haul increases

the time cycle of the haulage unit and reduces its hourly capacity; moreover, increase in depth adds to the power expended in bringing material out of the pit.

In many pit operations, the relation between depth and area prohibits the use of long, circuitous haulage routes for either locomotives or trucks. The difficulty is frequently overcome by retaining either unit to serve the shovel, but restricting its operation to the pit floor. Either delivers to the foot of an engine plane<sup>51</sup> or to an inclined field belt conveyor.<sup>84,103</sup>

Haulage in dry pits in which gravel is mined by hydraulicking parallels that for bank deposits. The haulage problem from dry pits using the top-benching system is comparatively simple. If a dragline is used, it is served either by cars and locomotives<sup>85,87,99</sup> or by motor trucks, and the haul is by the most direct route over a comparatively level roadway to the plant. The power scraper and slackline cableway are often built with their head towers integral with the treatment plant<sup>67</sup> and hence haul direct to the plant. If the head tower is not a part of the plant, locomotives and cars or trucks may be used.<sup>39,76,83</sup>

All material lying below the surface of the ground contains water. If the material is porous and the subsurface drainage good, the water may be present merely as moisture, but if the material is impervious and the drainage poor, the deposit may attain a saturated condition. Where subsurface drainage is insufficient to carry off all ground water to some point below the lower limit of the gravel, the deposit becomes a wet pit and must be worked as such. The method used in mining a wet pit depends largely upon the elevation of the water table, but the quantity flowing is also important. To clarify this point a discussion of the physical formation of pit deposits is necessary in order to visualize and understand the effect of a changing water level. The author has pointed out the necessity for observing the water level in test pits and also the rapidity with which they filled to that level after having been pumped dry. The subsurface flow can thus be gauged approximately.

Gravel deposits themselves may be either impervious or porous, depending upon the amount of clay or other impervious matter present to fill the voids between sand and gravel particles. The strata enclosing a gravel deposit may be impervious or porous or have open watercourses. Obviously, the rate of migration of ground water through both the gravel and surrounding strata will vary greatly. Those containing clean loose gravel with little fine sand, and surrounded by strata of open structure, will hold little water. Those containing large quantities of clay or fine sand, and surrounded by impervious strata, will be saturated with water. Between these two extremes lies a large group of combinations affecting both water-level and subsurface flow. They are all wet pits, but their water content ranges from nearly dry to saturation and from a light to heavy flow.

The degree of porosity of the gravel and the material surrounding it determines the rate of subsurface water flow. This cannot always be determined by shallow test pits in and around the deposit. For example, a deposit containing considerable clay and with its upper part surrounded by clay, as shown by test pits, would indicate a condition favorable to a stable water level. However, open passages below or porous material surrounding the lower part of the deposit—not penetrated by test drilling—might easily upset such conclusions. Plans for dredging such a deposit would be economically fatal because of eventual but slow loss of water.<sup>26</sup>

The bottom-bench system for wet pits is limited to stripping overburden<sup>16</sup> or removing that portion of the gravel lying above the permanent ground-water level.<sup>88</sup> For either, the method is exactly the same and utilizes the same equipment as in bottom-benching dry pits.

Wet pits are commonly mined by top benching. The methods and equipment are the same as for dry-pit mining, except that an excavator crane equipped with clamshell or orange-peel bucket is frequently used. Ground water or the elevation of its surface in the pit or its rate of flow make little difference, as the dragline, power scraper, slackline cableway or crane excavates under water with comparative facility.

Overburden may be removed by bottom benching,<sup>43</sup> or it can be stripped by a separate single top bench.<sup>36, 43, 57, 69, 70</sup> With wet-pit operation, however, there is less need for separate stripping of even thick overburden<sup>27</sup> because it can be caved into the water and washed fairly well as it is brought up with the gravel.<sup>37, 52, 89</sup> If necessary, the dragline can strip and cast to the water in the excavated pit beyond the gravel bench.<sup>91, 94</sup> The power scraper and slackline excavator can also strip and cast overburden to excavated areas.

In pits in which the water level is below the top of the gravel, the upper portion may be mined by top benching, the material being taken both from below and above the water in a single bench.<sup>54, 89</sup> In some deposits material lying above the water level may differ physically from that below, or it may be desired to mine that above and keep it dry. Multiple top benching is then the solution. The dragline removes the overburden first and casts it to the excavated pit. It next mines a second bench down to the water level and finally a third bench from below the surface. The power scraper and slackline cableway also may be used for this purpose, but the area of each bench will be increased.<sup>102</sup> The crane can restrict the amount taken from each bench to a single bucketload.<sup>40</sup> On the other hand, in stratified deposits containing clay, single-bench mining is often an advantage in wetting all material thoroughly and mixing that from different levels before it is sent to the plant.<sup>21</sup>

Dredging is primarily top benching, as the dredge removes material from below the surface of the water on which it floats. However, in a wet pit the water level is frequently some distance below the top of the bench.

hence, the dredge actually excavates from the bottom, as does a shovel in bottom benching. For this reason, dredging has been classed as intermediate between bottom and top benching. Wet pits may be mined by any type of dredge,<sup>15</sup> although the hydraulic dredge<sup>40,41,43,57,78</sup> is by far the most common. Ladder<sup>72</sup> and clamshell dredges are sometimes used in wet pits, but the dipper dredge is seldom used for this type of deposit. It is of the utmost importance to know the rate of subsurface flow of ground water before installing a hydraulic dredge in a wet pit, as the dredge ordinarily pumps 9 cu. yd. of water with 1 cu. yd. of sand and gravel removed. This water must be supplied by subsurface flow through the gravel and surrounding strata. With either type of dredge the method, whether classed as bottom or top benching, is carried as a single bench. Hydraulic giants are frequently mounted on dredges<sup>43</sup> and the stream is played on the bank to cave it ahead of the dredge.

Haulage equipment for wet pits is the same as that for dry pits, when bottom-bench methods are used.<sup>43,68</sup> If dry-land excavators are used with the top-bench method, the haulage methods and equipment are also similar to those for dry pits, because only the excavator bucket enters the water and it delivers its load to the haulage unit or plant on land.<sup>36,42,53,76,101,102</sup> Hydraulic dredges frequently use the dredging pump to do double duty—to dredge and to deliver dredged material through pipe lines to the plant on shore.<sup>40,43,47,53,57,58</sup> In compacted material a dragline has been used to break up and excavate the gravel and deliver it to the suction of a dredge pump for delivery to the plant.<sup>105</sup> If the distance or elevation is too great for the dredge pump to handle the discharge, booster pumps often are inserted in the discharge line.<sup>79</sup> In some recent installations the dredge pump is used to pump material through a floating pipe line to a shore station where the gravel and sand are dewatered by screens or other means and then carried to the plant by field conveyer belts,<sup>93</sup> trucks or cars. For haulage from distant points in large pits towboats and barges<sup>41</sup> are used. The barges are seldom of the self-unloading type but depend upon shore equipment to transfer their cargo to further conveyance or plant on shore. Shore equipment for this purpose is usually a clamshell crane mounted on a permanent or crawler base. A few self-unloading barges are equipped with conveyer belts, which receive their loads from the bottoms of cargo hoppers and discharge to other conveyers on the dock.<sup>41</sup> The haulage equipment used to serve ladder dredges is always some type of towboat and barge.<sup>72</sup> Although frequently used for placer mining in wet pits and for marine gravel deposits, the ladder dredge is not often employed for gravel production from this type of deposit.

Marine deposits have similar characteristics, irrespective of the body of water under which they lie, but for convenience they are subdivided into river, lake and ocean deposits. Subaqueous mining is the most

common method of recovering gravel from marine deposits. There are some exceptions in river beds, as will be noted.

Excavation equipment for this method of mining is restricted to some type of dredge, but its design may differ materially from a similar type used in wet pits.<sup>88</sup> For illustration, dredges used in marine deposits must be designed to withstand floods or wave action during storms and to operate in stream or tidal currents and under changing depth conditions due to the rise and fall of tides. For ocean deposits, they must be designed to resist the corrosive action of salt water. Haulage equipment must, in general, be seaworthy, and the self-propelled barge is used extensively. Where currents are troublesome, dikes sometimes are built between islands or between an island and the river bank, behind which dredges may mine the river bed under conditions simulating those of wet pits.<sup>62</sup> Similarly, a lake or ocean dredge may abandon the subaqueous bed and cut its way from open water through the beach into a shore extension of the bed.<sup>1</sup>

River beds and small islands may be mined by top benching with power scrapers<sup>104</sup> or slackline cableways<sup>106</sup> located on the river bank, by dredges floating on the stream<sup>38,98</sup> or by both.<sup>107</sup> Dredges used in river work are often called upon to mine beds at widely scattered points. Because of the necessity for this extended mining range there is greater preference for towboats<sup>55</sup> and barges as the transportation medium. In addition, many dredges used for river work are designed to include washing and screening equipment and thereby avoid hauling waste products over long distances.

Although dredging in a river bed is technically top benching, the removal of an island deposit by dredging might be classed as bottom benching. Therefore, to avoid confusion, the term "top benching" used in connection with marine deposits will be limited to excavation by power scrapers or slackline cableways operated from the river bank.

Top benching by these two units may be further subdivided, depending upon whether the excavator is permanently located on the bank<sup>37</sup> or is free to move. Stationary equipment is usually built with the head tower integral with the washing plant. Permanent plants are limited in operating life to the time required to exhaust the bed or bar within reach of the scraper or slackline, except that in some rivers the area excavated is refilled by new gravel brought down by the river itself during times of flood.<sup>37,104</sup> At other places, when the permanent plant has exhausted material within reach, the scraper may be left intact and a dredge installed to dig distant bars and deliver by barge to the scraper.<sup>107</sup> If the excavator is portable, the scraper or slackline is moved to a new location when the bar is exhausted, but auxiliary haulage units must be installed to serve the excavator and deliver to the plant.<sup>104,106</sup>

Draglines have not been mentioned in connection with river-bed excavation because of their limited reach from the river bank. They may, however, be used to mine an island deposit and load barges moored to the bank. To warrant such equipment, the island would have to be large; the operation would then approach wet-pit mining.

Customarily, river bars are dredged selectively. The dredge is frequently alternated between several bars, according to the type of material needed for the market. The hydraulic dredge is the most popular for river deposits. Where large tonnages are required from extensive bars the ladder dredge<sup>71</sup> rivals the hydraulic or pump dredge. Grapple or clamshell dredges<sup>71,107</sup> are used principally in excessively deep water or where boulders or logs would make the use of hydraulic or ladder dredges difficult. Dipper dredges have been used for gravel mining in only a few instances and were selected because of the large number of boulders in the deposit.<sup>15,73</sup>

When power scrapers or slackline cableways are used to top-bench river deposits, the excavator itself acts as the haulage unit, unless it is portable and requires the service of other equipment to complete delivery to the washing plant.<sup>104,106</sup> This auxiliary equipment may consist of cars, trucks or conveyer belts, which usually operate over fairly level surface.

Where permanent plants have exhausted material within reach and actual excavation has been delegated to supplementary dredges, transportation usually is by towboat and barge, but it may be by pump and pipe line if the dredge is hydraulic. Many of the barges used are self-dumping;<sup>107</sup> they are loaded at the dredge, towed to the site of the power scraper or slackline, and dumped to the river bed, from which the scraper transfers the gravel to the shore plant. If the dredge is hydraulic and the distance not too great, it may pump gravel directly to a point within reach of the scraper.

In opening a new bar a hydraulic dredge may deliver directly to the shore plant. If the bar is extensive and the resistance grows too great for the dredge pump to both transport and elevate to the shore plant, a second dredge may be placed at the shore near the plant. The original dredge, relieved of elevating the gravel, can pump directly to the second dredge, which thus acts as a booster; or to water storage within reach of the second dredge.

Various other types of transportation may be combined.<sup>106</sup> For example, at one plant a hydraulic dredge excavates and delivers gravel to a sump from which it is elevated by a clamshell crane to a washing plant, transferred from there by belt conveyer to an aerial tram, and carried by the tram to the shore plant.<sup>52</sup> Ladder, grapple and dipper dredges require barges as haulage equipment. In some instances, the barges are self-propelled and fitted with self-unloading equipment. Both



dredges and haulage units used in mining river deposits frequently are required to operate in very shallow water.<sup>98</sup> For this purpose, pump dredges have been designed to work in places where the maximum draft was 2 ft. or even less. Barges, however, seldom are used in less than 4 ft. of water. Ladder dredges are used for lake or ocean deposits only under very sheltered conditions, hydraulic dredges are common, grapple and dipper dredges are used mostly for channel and harbor excavation, tugs are seagoing, and barges are usually the round-bottom, full-hopper type.

#### PREPARATION OF SAND AND GRAVEL FOR MARKET\*

The methods and equipment necessary to prepare sand and gravel for the market vary widely, according to the method of excavation (or mining) employed, the nature of the bank material, and the demands of the

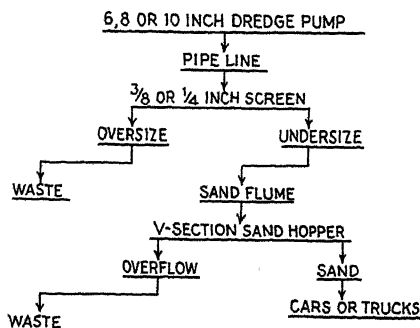


FIG. 5.—RIVER-SAND DREDGING.

market in which the product must be sold. The simplest plants are those that produce sand with little or no gravel. Most of these are erected to treat material pumped from a river bottom. A typical flowsheet (Fig. 5) would include a pump dredge, a screen to remove trash and gravel and a hopper to receive the sand that has passed the screen. This hopper is of V-section and usually there are three or four openings toward the bottom, closed by gates that are operated by hand. The flume that passes over the hopper has three or four openings in the bottom and occasionally is operated to produce a rough classification.

Where gravel is present the oversize of the screen, instead of going to waste, may go to screens to make one or more sizes of gravel. In the simplest plants of this kind (Fig. 6) all the screens are flat.

With larger tonnages and a considerable proportion of gravel, a different arrangement is necessary. A good flowsheet (Fig. 7) for such conditions begins with a surge box that receives the pump discharge.

\* Contributed by Edmund Shaw, Consulting Engineer, Los Angeles, Calif.

# SAND AND GRAVEL

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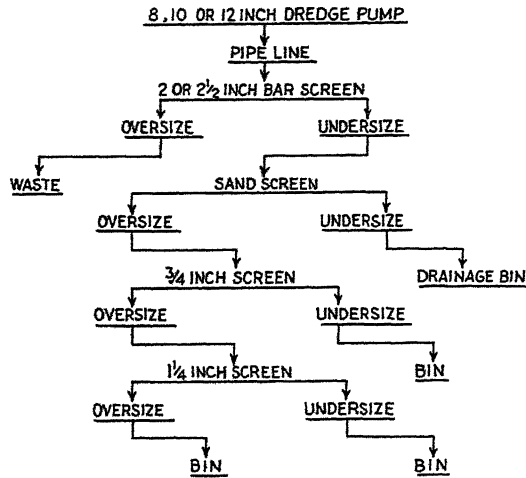


FIG. 6.—RIVER-DREDGING PLANT WITH STATIONARY GRAVEL SCREEN.

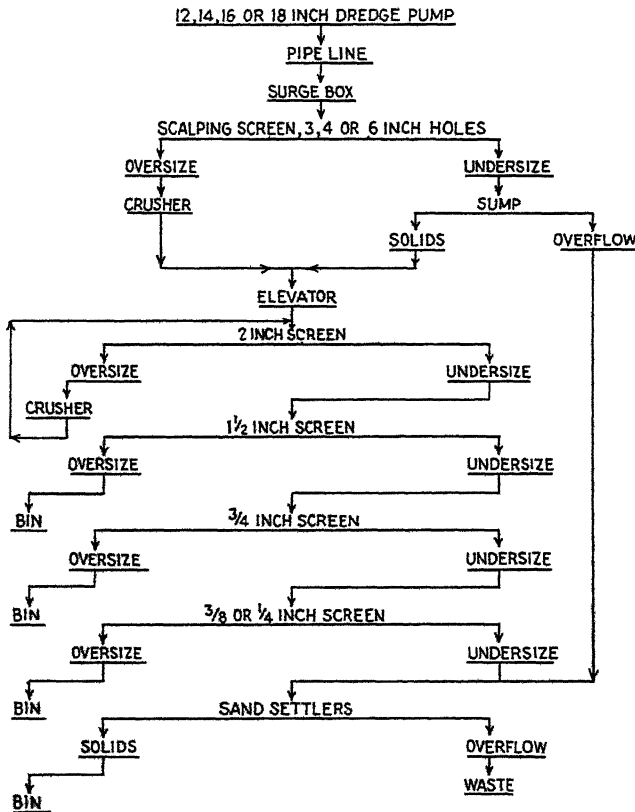


FIG. 7.—LARGE DREDGING PLANT WITH CRUSHERS.

This is followed by a screen or grizzly that removes the large cobbles and occasional boulders that go to a crusher. The sand, gravel and water go to a sump from which the solids are excavated by a heavy-duty chain-and-bucket elevator, which lifts them to a series of screens. The crushed material may fall from the crusher into the sump and be elevated with the sand and gravel, or it may be sized and sold as a separate product. The advantage of this design is that elevating the sand and gravel to the screens is cheaper than raising it to the screens by the dredge pump. There are several variations of this flowsheet. Where there are many large pieces the pump may discharge to a large sump above an inclined tunnel containing a track on which runs a skip. This skip has an automatic discharge and reverse, although the loading is usually done by a manually operated gate. Another variation is to discharge the material

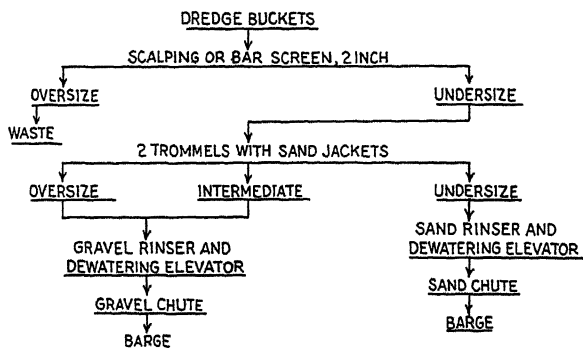


FIG. 8.—LADDER DREDGE, ALL MATERIAL ON HULL.

to a pile under water from which it is taken by a slackline cableway to the top of the plant. Where there is much crushing to be done a chute returns the oversize of the first screen to a secondary crusher.

Large dredges have the screening and washing plant on the hull of the dredge. The washing and screening plant is usually simple, the refinements in sizing and regrading being given the material in a plant on shore (Fig. 8).

A shore plant having no connection with the dredge except by barges is a link between the dredge plants and those that treat the material dry. The actual plant is not different from a dry-land plant except in the method of feeding, which is usually by means of a boom derrick with a clamshell bucket.

Of dry-land plants, that which is designed to go with the slackline-cableway excavator is probably most used (Fig. 9). The commonest design has a grizzly at the top on which the cableway bucket discharges. The oversize pieces roll off the grizzly to a chute and thence to a crusher bin. The crusher is in closed circuit with a revolving screen, unless there is a secondary crusher. In that case, the crusher discharge goes to a

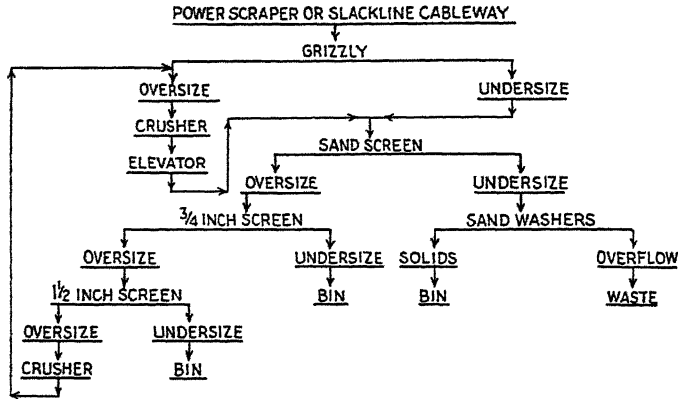
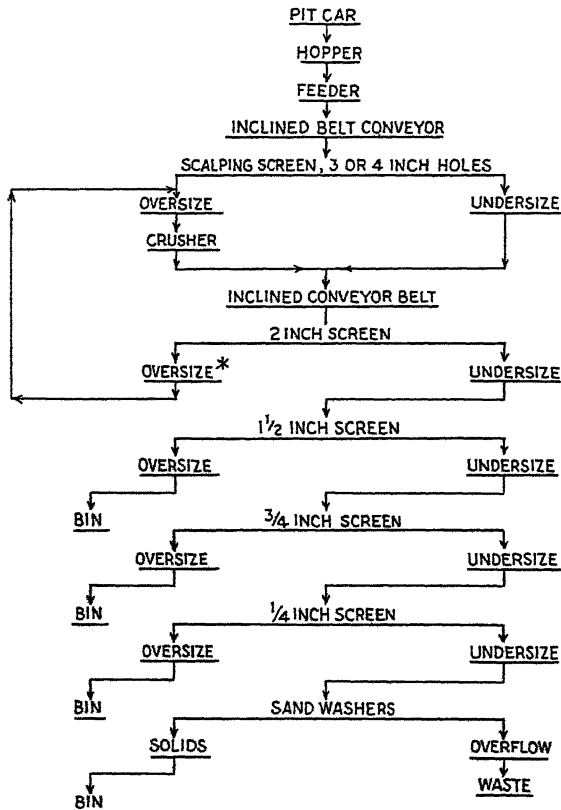


FIG. 9.—DRY-LAND PLANT WITH SCRAPER OR SLACKLINE.



\* WHEN THE AMOUNT OF 2-IN OVERSIZE IS LARGE, IT IS SENT TO ONE OR MORE SECONDARY CRUSHERS. IN A FEW PLANTS IT IS SOLD AS A SIZED PRODUCT

FIG. 10.—TYPICAL DRY-LAND PLANT WITH POWER SHOVEL, DRAGLINE OR POWER-SCRAPER MINING.

screen that is in closed circuit with a secondary crusher. The gravel-screening and sand-recovery devices are the same as in other plants.

The type of plant that probably is producing more tonnage than any other, except perhaps the large dredge plants, is that used with power scraper, power shovel or dragline mining of bank material (Fig. 10). Commonly, the shovel puts the bank material into a car, which is pulled into the plant and spotted over a track hopper. An inclined conveyer, which has its lower end under a gate below the hopper, takes the material to the scrubber or the first screen at the top of the plant. If the ground contains many large cobbles and boulders, an automatic skip may take the place of the conveyer. But a primary crusher on the ground level is a better arrangement. In this arrangement, the car usually is pulled to a level above the crusher and discharges over a grizzly above a hopper.

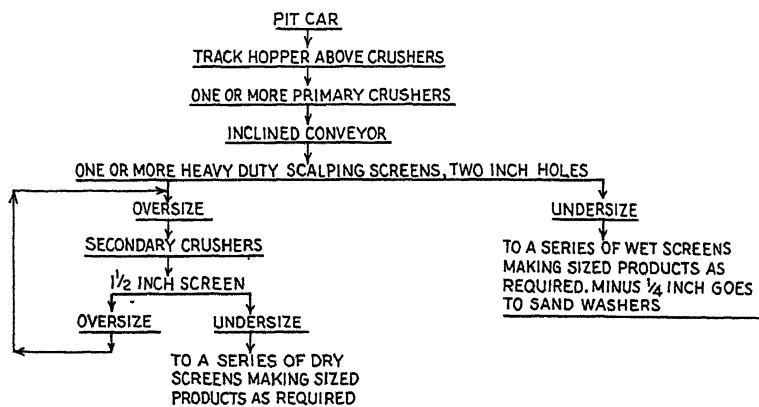


FIG. 11.—PLANT FOR DEPOSIT WITH MANY BOULDERS AND COBBLES.

Arrangements are usually made for recrushing the larger of the stone sizes, and sometimes the larger gravel sizes, either by a separate crusher or by returning to the secondary crushers, whenever the market demands more fine sizes.

The oversize of the grizzly goes to the primary crusher and the discharge of the crusher joins the undersize on the conveyer belt that feeds the screens. Where there is not too much oversize a favorite method today is to bring the material from the shovel by conveyer belt. The conveyer feeds directly to a screen or a grizzly above the primary crusher and a second conveyer takes everything to the screens and washers. Such plants are built for tonnages of 100 to 500 tons per hour. Perhaps 300 tons per hour would be about the average. There are many minor variations in the plant design, depending upon the location, the material and the designer. Many such plants are designed by machinery manufacturers and are planned to use the manufacturers' machines.

Certain deposits in the United States contain so many boulders and cobbles that the operation is that of a stone crusher with an incidental production of sand and gravel (Fig. 11). Such deposits are found in the

Middle West, formed from terminal moraines of the glacial era, and on the Pacific Coast. In some localities the crushed boulders sell at a higher price than gravel, so the material is split at the start into gravel, 2 or  $2\frac{1}{4}$  in. down, and cobbles and boulders that require crushing. The gravel and sand go by one conveyer to wet screens and sand washers and the crushed material to dry screens, so that there are really two plants. These are the largest and costliest plants in the industry, and the tonnage must be large in order to pay for them in a reasonable time.

### EQUIPMENT

Plant equipment consists of crushers, screens, sand washers and settlers and the necessary conveying and elevating machinery. Jaw crushers are used in the smaller plants because of their low first cost, simplicity, and the ease with which they can be repaired.\* They are also used as primary crushers in some large plants. Where the largest pieces run 14 to 16 in. in diameter or less, gyratory crushers are preferred for plants of more than 100 tons per hour. They are usually of the types built for crushing quarried rock, although some makers design with special attention to gravel crushing. Jaw crushers are rarely used for secondary crushing, although a double-jaw crusher has recently been placed on the market for secondary gravel crushing.† Cone crushers and types of gyratories that approximate them in design are most in favor. Rolls are occasionally found in plants in the far West.‡ A few plants have converted plain rolls to corrugated rolls by welding rods of a wear-resisting alloy across the faces.\* Independent drives and motors for the separate rolls are said to increase crushing efficiency by the differential effect.

A decade ago, most plants were equipped with revolving screens of the trommel or conical types. Today, inclined flat screens with different methods of vibration are much more in favor. The advantage of the revolving screen is that it is a good scrubber and washer, but in the opinion of some operators this is not so important as the high screen efficiency per foot, the ease with which the screen sizes can be changed, the ease with which repairs and replacements can be made and the saving in space and headroom. Some types of vibrating screens have mechanical vibration and others electrical. The shaking screens with a reciprocating motion, which once were out of favor, have returned to use by reason of

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\* E. Shaw: New Small Sand and Gravel Plants in the Los Angeles District. *Rock Products* (1934) 37, No. 10, 38.

† A Double Jaw Crusher. *Rock Products* (1936) 39, No. 1, 42.

‡ Arizona's Largest Washed Gravel Plant. *Rock Products* (Oct. 12, 1929) 32, 69.

better design.\* The vibrators are improvements over earlier types in that they give less wear on the screen cloth without decreasing the efficiency.

There are three systems of screening in sand and gravel operations. The first follows crushed-stone practice, taking out the oversize with the first screen and making successively finer products until the last or the sand screen. A second method is to take out the sand, with the accompanying water and clay, at first and to screen the gravel dry, except for the water furnished by the sprays in the screen. A third method is to split the feed by a screen that has openings about one-half of the maximum size, as 1 in. or  $\frac{7}{8}$  in. for a maximum of 2 in.<sup>66</sup> Each half is then sent to a separate line of screens.

Of revolving screens, the conical types appear to be most favored. The cylindrical types with one or more jackets make a very compact screening unit, and are useful on dredges, or wherever space must be conserved. But where there is room to put the bins for the products in series, a line of separate screens fits the design better.

The screening media are punched plate and woven mesh. Plates are punched in a variety of ways: square, round, triangular<sup>66</sup> and diamond-shaped holes. The last has been found the most efficient in some Pacific Coast plants. Steel wire is mostly employed for wire-mesh screens; manganese steel has considerable use for the coarser sizes. Stainless-steel wire-mesh cloth has given excellent results on the finer sizes.

Grizzlies (bar screens or grills) usually made of railroad rails are used at the head of many plants. Usually they are inclined and stationary but a few plants used revolving bar screens or disk grizzlies.

Washers are of two sorts. Those that have no overflow are called scrubbers, and their only function is to loosen the clay from the grains so that it can be carried away by a current of water in another machine. The most used scrubber is a plate-iron cylinder with "lifters" of angle iron, riveted to the inside, that lift the material and throw it over and over. The cylinder is set on an incline so that the contents flow through while being washed. An improved type has the cylinder set flat and works the material forward by blades placed in a spiral inside the cylinder.<sup>†</sup> A current of clean water is admitted at the discharge end and flows to the end where the feed is admitted. This both scrubs and washes, the latter by the countercurrent of clear water. A conical mill with no lifters is used as a scrubber in some plants.<sup>‡</sup> The grains in a mass with little water clean themselves by rubbing on one another. Log washers

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\* Reciprocating screens have been especially favored by the designers of plants for making aggregates for the great dams and viaducts recently built and building in the far West.

<sup>†</sup> The Hardinge scrubber.

<sup>‡</sup> So used successfully in plant of P. J. Weisel, Corona, Calif.

are successfully used to wash gravel in plants in the Middle West and the South.\* Washers pass the material through water to settle in a box or tank from which it is discharged as a thick pulp by a controlled orifice, or it is drawn out on drags or rakes or scoops mechanically operated.

Formerly sand cones were much used, devices that settled the sand in an inverted cone or pyramid, the discharge being from a gate at the apex, which was controlled by the weight or height of the sand in the cone. Today other types are more in favor. There are two kinds. In one, the current flows through a long box in which the sand settles and the water and clay flow away over a gate at the end. The settled sand is drawn out by a belt or chain with drags or by rakes operated by a reciprocating mechanism. The other kind has a round tank in which the sand is settled and the excavating is done by an inclined wheel with scoops. The inclination of the wheel is sufficient to bring the scoops above water at one point of their travel. Formerly it was enough if these devices produced a sand comparatively free from clay. Today they are set to throw out a certain portion of the finer grains of the sand, and are provided with adjustments by which the fineness of the sand discharged can be regulated. These are called classifiers and produce sands with fine and coarse particles to be afterward blended to make a predetermined grading. In some large plants very elaborate arrangements have been made to produce such sands, which are called "high-specification" sands.†

In earlier years the prepared material was largely produced in plants of capacities up to 500 tons per hour, located on railroads. This was stored in large yards fitted with cranes, conveyers, and other equipment for loading and unloading railroad cars, requiring a great deal of invested capital. The development of truck transportation made it possible to deliver sand and gravel to distant points at less cost than that of loading into railroad cars, freighting to a storage yard, and shipping by truck to the job. For markets too far to be reached readily from the main plant, it is becoming customary to erect portable, or readily movable, plants to furnish the material for a single job.‡ Some producers have two or more such portable plants working in addition to the main plant. This has caused a careful study of the design and operation of portable plants.

Many sand and gravel producers now sell a considerable part of their production as ready-mixed concrete. This simplifies the preparation as it reduces the number of sizes to be made and does away with much rehandling of the product.

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\* A. R. Amos and S. B. Patterson: Log Washers in the Aggregate and Flux Stone Industries. *A.I.M.E. Tech. Pub.* 679 (1936).

† A. Anable: Preparation of High-specification Sand at the Grand Coulee Dam. *A.I.M.E. Tech. Pub.* 715 (1936).

‡ Fort Peck Dam Aggregates Plants. *Rock Products* (Jan. 1936) 39, 84-85.



Shale, soft sandstone, coal and some other minerals are unfit for concrete aggregate, and gravel is not salable if it contains more than a small percentage of such particles. There are two ways of elimination: (1) by jigging,<sup>61</sup> and (2) by breaking down the soft minerals without fracturing the pebbles. Jigging is successful with minerals that have an appreciable difference in specific gravity. The method of breaking the soft pieces by throwing them by centrifugal force against the inside of a cylinder has been found to work well in some plants. Recently concentrating tables of the type used for cleaning coal have been successfully used to remove shale from gravel in sizes up to  $2\frac{1}{2}$  in. in diameter.

The following screens have been noted in use in gravel plants, although only a few of them are used in any one plant: 6-in., 4-in., 3-in.,  $3\frac{1}{2}$ -in., 2-in.,  $1\frac{3}{4}$ -in.,  $1\frac{1}{2}$ -in.,  $1\frac{1}{4}$ -in., 1-in.,  $\frac{7}{8}$ -in.,  $\frac{3}{4}$ -in.,  $\frac{5}{8}$ -in.,  $\frac{1}{2}$ -in.,  $\frac{3}{8}$ -in.,  $\frac{5}{16}$ -in.,  $\frac{1}{4}$ -in., 4-mesh and 8-mesh. These are all square holes, and each has its approximately round-hole equivalent. The sizes above  $2\frac{1}{2}$  in. are used only to make aggregates for the largest work, and the sizes between  $1\frac{1}{2}$  and  $2\frac{1}{2}$  in. are less used than formerly, the ordinary maximum sizes today being  $1\frac{1}{2}$  and  $1\frac{1}{4}$  in., with 1 in. in thinner sections. The tendency is to make fewer sizes, but custom makes it hard for producers to change, as their buyers demand certain sizes with which they are familiar. A more or less typical set of products might be:

1. Gravel,  $2\frac{1}{2}$  to  $1\frac{1}{2}$  in. used in concrete aggregate and bituminous paving.
2. Gravel,  $1\frac{1}{2}$  to  $\frac{3}{4}$  in. Same use as above.
3. Fine gravel,  $\frac{3}{4}$  to  $\frac{3}{8}$  in. Used as above and also in other kinds of road surfacing.
4. Roofing gravel,  $\frac{5}{8}$  to  $\frac{3}{4}$  in. or  $\frac{1}{2}$  or  $\frac{1}{4}$  or  $\frac{3}{8}$  in. to No. 4 mesh. Used in concrete, bituminous paving, road surfacing, roofing and industrial uses.
5. General purpose sand, or washed concrete sand,  $\frac{1}{4}$  in. down. With a fineness modulus of 2.50 to 3.40 and a good grading, this is the sand specified by Government engineers on some of the largest works. It is the ordinary concrete sand, and a large tonnage is also sold for making mortar and certain kinds of plastering.
6. Asphalt sand, 10 mesh down. It is usually a combination of several size gradations. It also has a considerable use in correcting coarser concrete sands.
7. Asphalt filler, 100 mesh down. It is used in bituminous mixtures to increase density.

The sizes as noted above are not market sizes but illustrate primary separation in the treatment plant. Market sizes are combinations of these primary divisions as discussed under "Specifications."

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\* F. Sinclair: Problem of Clean Sand and Gravel Solved by Concentrating Tables. *Rock Products* (1934) 37, No. 11, 26.

## TESTS

Comprehensive tests on samples can be made only in a properly equipped laboratory by trained personnel. Few operators are so equipped at their plants. Consequently, final testing is usually a matter for the commercial laboratory. It is not the function of this chapter to describe in detail the exact test methods of the laboratory, but field tests are discussed.

The value of a deposit may be roughly determined by various field tests. Should these indicate that the deposit has commercial possibilities, similar samples should be subjected to exhaustive laboratory tests. In only a few instances will it be necessary to have final tests made before a decision can be reached as to the commercial possibilities of the deposit. Preliminary testing is confined to such items as: (1) the specific gravity; (2) absorption; (3) the percentage of organic impurities present; (4) the percentage of clay and silt present; (5) the size gradation of sands and gravels; (6) the percentage of soft, friable and unconsolidated particles present; (7) the presence or absence of deleterious coatings.

The following field tests are suggested and corresponding laboratory tests as recommended by the American Society for Testing Materials are cited:

*Specific-gravity Tests.*—The terms commonly used in sand and gravel determinations are “apparent specific gravity” and “bulk specific gravity” (A.S.T.M. designation E-12-27).

The sample to be tested for specific gravity should first be ground to pass 200 mesh, then dried to constant weight in an oven at  $105^{\circ} \pm 5^{\circ} \text{C}$ . and weighed in air (A).

It is then immersed in water for 24 hr., removed and the moisture dried from the surface of the particles (called surface drying) and again weighed in air (B).

It is then immersed in water and weighed (C).

The apparent specific gravity can then be calculated by the formula

$$\text{Apparent specific gravity} = \frac{A}{A - C}$$

and the bulk specific gravity by the formula

$$\text{Bulk specific gravity} = \frac{B}{B - C}$$

*Absorption.*—For a more accurate laboratory method see A.S.T.M. designation C-127-36T and C-128-36T.

The absorption test is performed in determining the specific gravity:

$$\text{Absorption} = \frac{B - A}{A}$$

$$\text{Percentage of absorption} = \frac{B - A}{A} \times 100$$

*Organic Impurities.*—Organic impurities, such as leaves, twigs, peat and coal can be eliminated by burning. As low as 0.05 per cent of certain organic impurities if contained in aggregates is injurious to concrete.\* On the other hand, appreciably greater amounts of other organic impurities such as lignite have been permissible.†

A common field test for organic impurities used in the past consisted in recording the weight of the sample in air when dried at less than 110° F. to constant weight. The sample was then heated to 400° to 500° F. for about ½ hr., cooled and again weighed. The difference in weight was recorded as the loss in organic matter. However, small amounts of impurities would present such small losses by this method as to avoid accurate measurement, therefore the colorimetric test should be made as follows:‡

Fill a 12-oz. graduated prescription bottle with the sand sample to the 4½-oz. mark. Add a 3 per cent solution of sodium hydroxide to the 7-oz. mark. Shake thoroughly and let stand 24 hr. Observe the color of the clear liquid above the sand. Clear or light yellowish color indicates sand satisfactory for concrete so far as organic impurities are concerned. Straw color indicates sand suitable for unimportant concrete. Dark colors indicate sand unfit for concrete. Sand showing straw or dark color should be examined for lignite. If present, the sand should not be condemned on account of the colorimeter test until it is confirmed by mortar strength tests. The more accurate method of determining the percentage of organic matter present is described under the A.S.T.M. designation C-40.

*Clay and Silt.*—The following test has been proposed to the A.S.T.M. to replace the clay and silt test:

In determining the amount of minus 200-mesh material present, record the weight of the sample as dried in air to constant weight, as  $W_d$ . Place the sample in a pan in which it can be covered with water. Stir thoroughly and pour off the water, through a 200-mesh sieve, being careful that no sand is lost. Add more water, stir and pour off. Repeat until the water removed is clear. Dry the remaining sample, plus any accumulation on the 200-mesh sieve, to constant weight and weigh. Record this weight as  $W_c$ .

$$W_d - W_c = \text{weight of minus 200-mesh material removed.}$$

$$\frac{W_d - W_c}{W_d} \times 100 = \text{percentage of minus 200-mesh material.}$$

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\* D. A. Abrams: Effect of Tannic Acid on the Strength of Concrete. Structural Materials Research Lab., Lewis Inst., Chicago, *Bull.* 7 (1922) 27.

† Effect of Coal and Lignite in Sand for Concrete. *Proc. Amer. Soc. Test. Mat.* (1929) pt. 1, 828.

‡ A.S.T.M. Standards (1930) pt. 2, 154.

A similar method is followed by some operators and has the advantage of using larger samples, thus avoiding possible errors in sample reduction.

A small revolving screen ( $\frac{1}{4}$ -in. mesh) is mounted in a watertight wooden or metal box. The box is partly filled with water so that the material in the screen is submerged. The sample is then placed in the screen and revolved. The material retained on the screen will be gravel, and that passing through to the box will be sand, clay and silt. The latter is allowed to settle, and the water drained off. The sand, clay and silt are then subjected to the clay-silt test as previously described. This test has the advantage of showing the character of the clay and its effect in adhering to gravel, collecting fines, accumulating as balls, etc., under conditions simulating plant practice.

*Size Gradation.*—The sample is now ready for a sieve analysis. The first step in the test is a separation of the sand from the gravel. The dried sample from the clay and silt determination is weighed and the weight recorded as  $A$ .

The sample is then placed on a screen perforated with  $\frac{1}{4}$ -in. round holes and agitated to refusal; i.e., until no material will pass. The material passing the screen is weighed and the weight recorded as  $S$ . The material retained is weighed and recorded as  $G$ .

$$\begin{array}{ll} \text{Then} & S + G = A \\ \text{and} & S/A \times 100 = \text{percentage of sand} \\ & G/A \times 100 = \text{percentage of gravel} \end{array}$$

Sample  $S$  is then passed through the testing sieves for sizing. Various sets of sieve standards have been and are being used. For the purpose of uniformity it is recommended that the A.S.T.M. sieve standards as given under designations E-11 and E-17 be used. When so used, the sand sample of weight  $S$  will be sieved over and through sieves Nos. 4, 8, 16, 30, 50 and 100. The weight of the material passing each will be recorded in tabular form as follows:

Passing	Retained on	Weight
$\frac{3}{8}$	4	$t$
4	8	$u$
8	16	$v$
16	30	$w$
30	50	$x$
50	100	$y$
100		$z$
Total = $S$ = .....		( $S$ )

Then  $\frac{t}{A} \times 100$  = percentage passing  $\frac{3}{8}$  in. and retained on No. 4 sieve, etc.

Sample *G* will then be passed through screens having the following sizes of round perforations: 3-in., 2-in.,  $1\frac{1}{2}$ -in., 1-in.,  $\frac{3}{4}$ -in.,  $\frac{1}{2}$ -in. The weight of the material passing each and retained on the next smaller opening will be recorded in tabular form as follows:

Passing, In.	Retained On, In.	Weight
3	2	<i>o</i>
2	$1\frac{1}{2}$	<i>p</i>
$1\frac{1}{2}$	1	<i>q</i>
1	$\frac{3}{4}$	<i>r</i>
$\frac{3}{4}$	$\frac{1}{2}$	<i>s</i>
$\frac{1}{2}$	$\frac{1}{4}$	<i>t</i>
Total = <i>G</i> = . . .		<i>G</i>

Then  $\frac{o}{A} \times 100$  = percentage passing 3 in. and retained on 2 in., etc.

Local conditions may require the use of sieves and screens with openings other than the sizes shown, but the procedure for testing will be the same in any case.

For A.S.T.M. standard methods see designations C-41, D-7 and D-19.

The principal questions that arise in the mind of the operator in valuing a sand and gravel deposit after it has been demonstrated that sufficient volume is present are:

1. The percentage of sand present.
2. The percentage of fines that must be removed from the sand to make it marketable.
3. The percentage of fine gravel that must be wasted.
4. The percentage of gravel present.
5. The percentage of oversize that will require crushing.

With properly collected samples the preceding sieve tests will provide the requisite answers to these questions.

*Soft, Friable and Unsound Particles.*—This test applies to the gravel only and is made by what are known as the soundness and abrasion tests. Both of these tests require laboratory apparatus for accuracy. However, the experienced operator will be able to appraise closely the percentage of soft, friable and unsound particles present by a visual inspection, for which a portion of the sample is spread on a table and the deleterious particles separated and counted. This count compared to the total number of particles gives the percentage.

The sodium sulphate test is commonly used for a soundness test of material showing no soft particles on visual inspection. This test can be

made as follows: A saturated solution of sodium sulphate is prepared. It is important that this solution be saturated and kept in this condition. The gravel sample is then placed in this solution and allowed to stand for 18 to 24 hr. It is then removed, oven-dried and again immersed in the saturated solution for another 18 to 24 hr. This alternate soaking and drying is repeated five times. The test should be carried on in a room of which the temperature varies not more than 2° C. Particles of questionable durability crack or spall under this treatment. The A.S.T.M. designation for laboratory soundness test is C-88-31-T and C-89-31-T.

*Deleterious Coatings.*—Gravel particles are often coated with deleterious siliceous, calcareous or argillaceous materials, which if not removed cause unsound concrete. There is no particular test for these coatings other than the visual inspection of the washed sample. The apparatus mentioned under the clay and silt test (p. 706) will be valuable

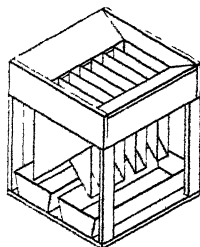


FIG. 12.—SAMPLE SPLITTER.

in facilitating inspection of the gravel under conditions simulating actual plant washing practice. In addition to the preceding tests, the material should be tested for abrasion loss. There is no practical field test for this, and it must be performed in a laboratory having the proper equipment. The A.S.T.M. designation for abrasion test is D-289-28T.

*Splitting Samples.*—Often it is desired to split the sample to have it tested by independent laboratories. In doing so, exceeding care must be taken, or erratic results will follow. Sand or fine gravel samples can be split without much danger of discrepancy, but as the material increases in size the difficulty of accurate cutting increases proportionately. The method commonly used is the cone-and-quarter method.

Better results are obtained in splitting samples of mixed sizes by the use of automatic samplers. These are boxes in which partitions are placed whereby material falling within one section will be diverted in one direction, while material in the next space will pass in another direction. In their use, the sample is shoveled onto the sampler, which automatically separates it into two representative portions. Fig. 12 illustrates a simple device for this purpose.

*Specifications.*—Evolution of technical design of concrete structures has produced many different specifications for commercial sand and

TABLE 2.—Group A, Sizes of Coarse Aggregates<sup>a</sup>  
(Crushed Stone, Gravel and Slag)

Size No.	Nominal Size Square Openings, In.	Approximate Size Round Openings, In.	Amounts Finer than Each Sieve (Square Openings), Per Cent by Weight															
			4"	3½"	2½"	2"	1½"	1"	¾"	½"	⅜"	No. 4	No. 8	No. 16	No. 50	No. 100		
			Primary Sizes															
1A . . . . .	2-3½	2½-4½	100	90-100	100	0-15	90-100	35-70	0-15	0-15	0-15	0-15	0-5					
2A . . . . .	1-2	1½-2½																
3A . . . . .	¾-1	¾-1½																
4A . . . . .	No. 4-½	¾-¾																
5A . . . . .	0-No. 4 mesh	0-¾																10-30
Combined and Modified Sizes																		
12A . . . . .	1-3½	1½-4½	100	90-100	100	25-60	90-100	0-15	0-15	0-15	0-15	0-5						
23A . . . . .	¾-2	¾-2½			100	90-100	95-100	20-55	20-55	10-30	0-5	0-5						
234A . . . . .	No. 4 mesh-2	¾-2½			100	95-100		35-70	35-70	0-10	0-5	0-5						
34A . . . . .	No. 4 mesh-1	¾-1½					100	90-100	90-100	25-60	0-10	0-10						
345A . . . . .	0-1	0-1½					100	90-100	90-100	30-65	10-25	10-25						0-10
45A . . . . .	0-½	0-¾							100	90-100	0-5	0-5						0-20
5A (Mod) . . . . .	No. 4-1	¾-1½					100	90-100	40-75	5-20	0-5	0-5						
4A (Mod) . . . . .	No. 8-½	¾-¾							100	90-100	40-70	5-25	0-5					
5B (Mod) . . . . .	No. 8-¾	¾-¾								100	90-100	10-30	0-8					
G <sup>b</sup> . . . . .	No. 50-1½	No. 50-1½					100	80-100	80-100	50-80	20-40	15-35	5-25	0-10			0-2	
G <sup>c</sup> . . . . .	No. 8-1½	No. 8-1½					100	65-100	65-100	35-75	10-35	0-10	0-5					
G <sup>d</sup> . . . . .	No. 4-1½	¾-1½					100	60-95	60-95	25-50	0-15	0-5						

<sup>a</sup> It is not intended that sizes from both groups (see group B, Table 3) will be adopted in any one locality. Each marketing area should base its specifications on the group best fitting local conditions and leading to the least change in the current specifications.

<sup>b</sup> The requirements for grading depend upon the percentage of crushed particles in gravel. Size G1 is for gravel containing 20 per cent or less of crushed particles; Size G<sub>2</sub>, for gravel containing more than 20 per cent and not more than 40 per cent of crushed particles; Size G<sub>3</sub>, for gravel containing crushed particles in excess of 40 per cent.

TABLE 3.—Group B, Sizes of Coarse Aggregates<sup>a</sup>  
(Crushed Stone, Gravel and Slag)

Size No.	Nominal Size Square Openings, In.	Approximate Size Round Openings, In.	Amounts Finer than Each Sieve (Square Openings) Per Cent by Weight														
			3½"	3"	2½"	2"	1½"	1"	¾"	½"	¾"	No. 4	No. 8	No. 16	No. 50	No. 100	
Primary Sizes																	
1B.....	2½-3½	3-4¼	90-100 <sup>c</sup>	85-70	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15
2B.....	1½-2½	1¾-3		100	90-100	85-70	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15
3B.....	¾-1½	¾-1¾				100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100
4B.....	¾-¾	¾-¾															
5B.....	No. 4 mesh-¾	¾-¾															
6B.....	0-No. 4 mesh	0-¾															10-30
Combined and Modified Sizes																	
12B.....	1½-3½	1¾-4¼	90-100 <sup>c</sup>	100	85-70	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15	0-15
23B.....	¾-2½	¾-3			100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100
34B.....	¾-1½	¾-1¾															
3A(Mod)....	No. 4-1	¾-1¾															
34B.....	No. 4-1½	¾-1¾			100	95-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100	90-100
45B.....	No. 4-¾	¾-¾															
45B.....	0-¾	0-¾															
45B.....	0-¾	0-¾															
5B(Mod)....	No. 8 mesh-¾	¾-¾															
45B(Mod)....	No. 8-¾	¾-¾															
G1 <sup>b</sup> .....	No. 50-1½	No. 50 mesh-1½															
G2 <sup>b</sup> .....	No. 8-1½	No. 8-1½															
G3 <sup>b</sup> .....	No. 4-1½	¾-1½															

<sup>a</sup> It is not intended that sizes from both groups (see group A in Table 2) will be adopted in any one locality. Each marketing area should base its specifications on the group best fitting local conditions and leading to the least change in the current specifications.

<sup>b</sup> The requirements for grading depend upon the percentage of crushed particles in gravel. Size G1 is for gravel containing 20 per cent or less of crushed particles; Size G2, for gravel containing more than 20 per cent and not more than 40 per cent of crushed particles; Size G3, for gravel containing crushed particles in excess of 40 per cent.

<sup>c</sup> 100 per cent finer than 4 inches.



TABLE 4.—*Typical Uses for Group A Sizes*

Typical Uses	Recommended Sizes												
	1A	2A	3A	4A	5A	19A	23A	294A	34A	344A	45A	8A (Mod)	4A (Mod)
Water-bound macadam:													
Coarse aggregate. . . . .	x	x			x	x							
Filler. . . . .													
Black base:													
Coarse aggregate. . . . .		x											
Choke. . . . .				x									
Bituminous macadam (penetration type):													
Coarse aggregate. . . . .		x											
Choke. . . . .				x									
Seal. . . . .													
Bituminous road mix:													
Mixing course. . . . .													
Choke. . . . .													
Seal. . . . .													
Drag-leveling courses:													
Leveling course. . . . .													
Seal. . . . .													
Bituminous surface treatments:													
Cold. . . . .													
Hot. . . . .													
Bituminous plant mixes <sup>a</sup> :													
Traffic-bound surfaces. . . . .													
Stabilized surfaces. . . . .													
Portland cement concrete. . . . .													
Railroad ballast:													
Stone or slag. . . . .		x					x						
Gravel. . . . .													
Roofing granules. . . . .													
Sewage filtration. . . . .	x	x		x									

<sup>a</sup> For plant mixes the aggregate should consist of appropriate sizes selected from Table 2 or Table 3 combined with suitably graded fine aggregate.

TABLE 5.—*Typical Uses for Group B Sizes*

Typical Uses	Recommended Sizes																		
	1B	2B	3B	4B	5B	6B	12B	23B	34B	3A (Mod)	34.5B	45B	456B	50B	5B (Mod)	45B (Mod)	G1	G2	G3
Water-bound macadam: Coarse aggregate...	x	x				x	x												
Filler...		x																	
Black base: Coarse aggregate...				x															
Choke...																			
Bituminous macadam (penetration type): Coarse aggregate...		x																	
Choke...				x															
Seal...																			
Bituminous road mix: Mixing course...									x										
Choke...																			
Seal...																			
Drag-leveling course: Leveling course...																			
Seal...																			
Bituminous surface treatments: Cold...																			
Hot...																			
Bituminous plant mixes* Traffic-bound surfaces...																			
Stabilized surfaces...																			
Portland cement concrete...																			
Railroad ballast: Stone or slag...			x																
Gravel...																			
Roofing granules...																			
Sewage filtration...	x	x																	

<sup>a</sup> For plant mixes the aggregate should consist of appropriate sizes selected from Table 2 or Table 3 combined with suitably graded fine aggregate.

gravel. These have been based largely upon the personal experience of the designing engineer. Raw materials vary considerably in different parts of the country and as a result specifications evolved in practice have been many and varied. The three aggregate associations have attempted to coordinate the requirements of engineers and set up standard specifications. For this purpose the National Crushed Stone, the National Sand and Gravel and the National Slag Associations set up a Joint Technical Committee. This committee enlisted the cooperation of the Division of Simplified Practice of the National Bureau of Standards in bringing about a general acceptance of standard sizes. Tables 2 and 3 show the recommended sizes and Tables 4 and 5 show typical uses for each of the proposed sizes as determined by the committee. The sizes are based on sieves with square openings and are divided into two groups, A and B, each of which is subdivided into "primary" and "combined and modified" sizes. Tables 1 and 2 also give the approximately equivalent round-opening limitations for sizes.

For further details concerning recommended standard size specifications, the reader is referred to the Division of Simplified Practice, National Bureau of Standards, Washington, D. C.

Specification requirements as to the percentage of organic impurities, clay or silt, soft, friable or unsound particles present are determined by local conditions and have not been nationally standardized. Detailed information regarding the extent of present standardization may be obtained from the National Sand and Gravel Association, Washington, D. C.

#### MARKETING, USES AND PRICE

Sand and gravel are found in almost every county in the United States, hence operations are less restricted by location of raw materials than in almost any other branch of the mineral industry. At the present rate, our reserves are being depleted by more than one hundred million tons per year. This would be infinitesimal if all the area known to contain sand and gravel were composed of commercial deposits. Much of this area is known to be patchy, as small deposits of good material are mixed with larger deposits of low-grade material. Also, much of it is at too great a distance from consuming centers.

It is significant that during 1932 Class I railroads received \$0.81 for each ton of sand and gravel transported, whereas the average value of sand and gravel as reported by producers was \$0.53 f.o.b. plant.\* This aptly illustrates the difficulty of producing sand and gravel for other than purely local markets.

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\* H. H. Hughes and E. R. Phillips: Sand and Gravel. Minerals Yearbook, U. S. Bur. Mines (1932-33) 610.

Somewhat more than one-half the sand and gravel produced from commercial operations is consumed in concrete construction, either highways or buildings. That produced from noncommercial plants goes largely to secondary highways, used with some type of binder for low-cost roads.

Highway and building construction is, then, the principal market for these materials. Until recently, street and building construction was concentrated largely in centers of population and highways designed to connect such centers, and large central producing plants were erected on

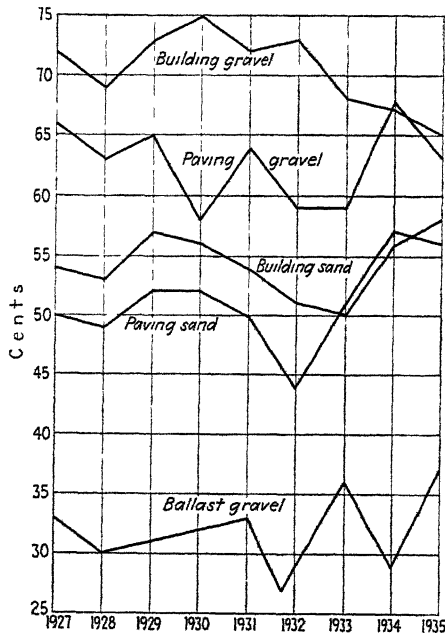


FIG. 13.—SAND AND GRAVEL PRICES PER SHORT TON F.O.B. PLANT.

near-by deposits. Cities and towns have completed much of their elemental street and local highway construction and during the depression years building construction declined abnormally. Thus, the centers of consumption have temporarily moved to intermediate points, away from the centers of population. The separation of these centers has increased haulage distances and with increasing freight rates has made it difficult for the large plant to compete with small portable plants set up on deposits in closer proximity to the new consuming centers. Hence, the industry as a whole has recently had a decided trend toward decentralization.

Considerable quantities of both sand and gravel are used in railway ballast and for fills in railway right of way. Owing to the poor financial condition of the railroads, road-bed maintenance has been neglected in

recent years, but with the return of better business conditions, and the need for better road beds to carry the new high-speed trains, this market has greatly improved.

Sand is prepared and sold to supply many other more specialized markets, but a separate chapter has been devoted to these special sands therefore they will not be discussed here.

*Price History.*—Fig. 13 gives a graphic history of the price range for sand and gravel since 1927, as taken from *Mineral Industry* and from the Minerals Yearbook of the U. S. Bureau of Mines.

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## CHAPTER XXXIX

### SLATE

BY CHARLES H. BEHRE, JR.,\* MEMBER A.I.M.E.

**S**LATE (*Thonschiefer*, German; *ardoise*, French) is a rock, an aggregation of mineral grains, occurring in nature, which is used for roofing and other special purposes. It is finely granular and crystalline and generally derived from an originally clayey bed or series of beds. As a result of its having been put under side pressure, this material now has its particles elongated and flattened so as to yield a very good rock cleavage along the flat surfaces of the minerals, a feature entirely independent of the original bedding in the rock.† Slate differs in varying degrees from most other common rocks (such as limestone, sandstone, granite and "trap") in chemical and in mineral composition. Its most characteristic feature, however, is its tendency to fracture along a series of very nearly parallel and very closely spaced smooth planes, called cleavage planes. This property of cleaving, above all else, gives slate its industrial value.

#### ORIGIN AND MODE OF OCCURRENCE

Most of the features of slate might be anticipated from its origin. Being a rock rather than a mineral, its composition may vary considerably without impairment of value. Much of the material of which slate is now composed was once deposited as clay on the bottom of seas, lakes or oceans. Subsequently these clayey strata were compressed from the sides by the same kind of lateral pressure that folds rock strata into mountain ranges, the minerals of the clay being changed or metamorphosed. In the chemical reaction involved, new and essentially plane-shaped minerals belonging to the mica groups were developed. Other minerals were formed but micas generally predominated. These were oriented so that their two longer dimensions are approximately at right angles to the last effective direction of pressure. Since the crystals are thus subparallel, fractures can be developed easily along their surfaces, much as cards may be separated by inserting a knife blade between them.

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† This is the gist of the definition adopted by the Committee on Slate of the American Society for Testing Materials; the original definition also lists the important and accessory minerals.

For the foregoing reasons, slate is found in noteworthy amounts only where (1) the original strata were largely clayey and (2) where there has been sufficient earth folding to induce recrystallization. Hence, for example, no commercial slate is found in the central Mississippi Valley; shales in that region have nowhere been sufficiently intensely folded to yield slate.

#### COMPOSITION AND PROPERTIES

*Chemical Composition.*—Chemically, slates are generally fairly uniform. Typical analyses given by Hirschwald<sup>11</sup> are:  $\text{Al}_2\text{O}_3$ , 12 to 26 per cent;  $\text{MgO}$ , 1.07 to 5.47;  $\text{SiO}_2$ , 35 to 59;  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ , 0.53 to 5.98;  $\text{CaO}$ , 0.14 to 17.75;  $\text{CO}_2$ , 0.11 to 11.81. In addition  $\text{FeO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{MnO}$ ,  $\text{BaO}$ ,  $\text{Li}_2\text{O}$ ,  $\text{P}_2\text{O}_5$ ,  $\text{SO}_3$ ,  $\text{ZrO}_2$  and  $\text{H}_2\text{O}$  may be present,\* generally in minor amounts. Of the constituents mentioned, much of the silica is combined with alumina and with basic oxides to form complex silicates (represented by minerals of the mica and chlorite groups). The carbon dioxide is a constituent of carbonates. Other representative analyses are given by Dale,\* Hirschwald,† and Behre.‡

Of interest is the preponderance of potassa over soda in the analyses, probably the result of base exchange and selective adsorption.<sup>18</sup>

*Mineralogical Composition.*—From the utilitarian viewpoint, the mineralogical is of more interest than the chemical composition, for mineralogy rather than chemistry determines perfection of cleavage, insulating qualities and strength. The chief minerals in slate<sup>23</sup> are present in the following quantities: muscovite, 38 to 40 per cent; chlorite, 6 to 18; quartz, 31 to 45; hematite, 3 to 6; rutile, 1 to 1.5. Other common minerals are the carbonates (calcite, dolomite, siderite and mixtures), kaolinite or related minerals (especially if the slate is not highly metamorphosed), graphitic carbon, biotite, various feldspars, andalusite, pyrite and magnetite. Dale gives a much longer list\* but many of the minerals mentioned by him are very rare or are primary and adventitious to any rock derived from sedimentary rock.

*Properties.*—As a structural material slate has properties such as hardness, crushing strength and density, in common with other building stones. Certain properties of particular significance in slate are discussed in the following paragraphs.

*Color.*—The consumer's taste in color of roofing slate varies greatly with time and locality, therefore color is very important commercially.

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<sup>11</sup> References are at the end of the chapter.

\* Ref. 8, 18, 23, 50-53.

† Ref. 11, 601-606.

‡ Ref. 3, 174 and 375.

Slates may be deep brick red, grayish purple, olive green, gray-green, dull bluish green, brownish (owing to stains of hydrous ferric oxide), or mottled in various combinations of these colors. The most common color is a bluish gray, "slate gray." Not all bluish gray slates, however, show the same tint; some have a silvery, light gray tone; others are almost black.

Color differences are due to presence of certain mineral constituents, such as hematite (ferric oxide) in red slates, ferrous compounds and

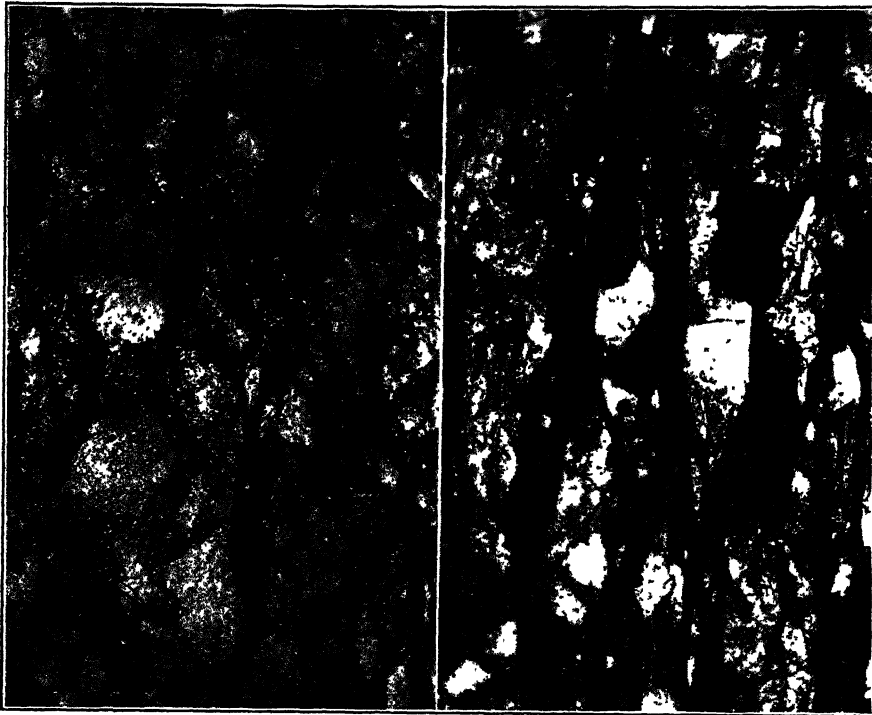


FIG. 1.—PETROGRAPHIC SECTIONS. ORIGINAL MAGNIFICATION 200, REDUCED  $\frac{3}{8}$  IN REPRODUCTION.

Section *a* is cut at right angles to cleavage and to grain; section *b* is in the plane of the grain. Note more pronounced elongation of dark, hairlike mica flakes in the section in the plane of the grain.

chlorite in deep green slates, sericite in light green and gray slates, and graphitic carbon in black slates. Since the original mud from which the slate was made was deposited in layers of varying composition, and since the cleavage pieces or blocks of the slate break *across* the bedding (as explained below), individual blocks may show bands differing in composition and hence in color. The most characteristic contrast is between the lighter and the darker, carbon-rich bands or "ribbons."

The color of slates is not always "permanent," and even exposure to the weather for 20 years may result in a change in color designated as "color-aging"\* to distinguish it from the steady disintegration of several centuries, which affects slate as it does any other stone. Most "color-aging" is probably due to alteration of iron sulphides, of iron oxides,† of carbon and carbon compounds,‡ and especially of the most soluble and least resistant minerals, the carbonates, notably when these contain iron.§ Thus it may safely be said that most color-aging effects are due to changes in ferruginous minerals and are not generally deleterious to structural use.

*Cleavage and Grain.*—There is still uncertainty as to the various theories for the origin of cleavage. In recrystallization of shale to slate

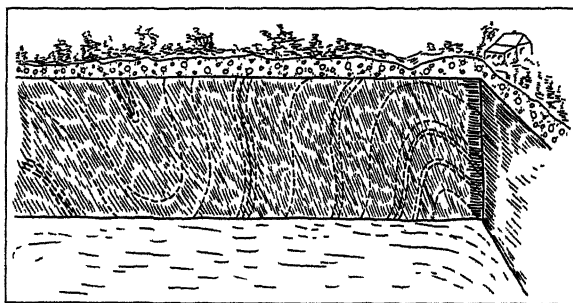


FIG. 2.—STRUCTURAL FEATURES OF SLATE AS SEEN IN QUARRY WALL, EUREKA QUARRY, SLATINGTON, PA.

Two adjacent folds are outlined by the bedding. The cleavage inclines steeply to right, generally parallel to axes of folds.

the newly formed platy minerals, especially mica, tend to be arranged approximately at right angles to the deforming pressure. Some minerals, such as quartz, not capable of this sheetlike elongation, yield to a linear or pencil-like but parallel elongation in planes parallel to the mica "sheets." In consequence, thin sections of slate show varying characteristics, depending upon their orientation (Fig. 1).

The parallelism of the pencil-shaped crystals, like quartz, makes it possible also to break slate at right angles to cleavage; such less regular planes of fracture are the grain planes. Grain planes, therefore, are generally normal to the cleavage planes. As a rule the cleavage planes are parallel to axes of the folded beds (Fig. 2). For a given region the trend

\* Ref. 3, 19.

† Ref. 8, 53.

‡ Ref. 11, 618.

§ Ref. 8, 56 and 140; ref. 3, 20-21.

or strike of cleavage and of grain is fairly constant, varying as a rule not over  $10^{\circ}$ .

*False Cleavage, Shear Zones and Related Features.*—If the cleavage itself is later deformed, it may show wrinkling or fracturing—minute, closely parallel wrinkles or cracks on the cleavage surface of the slate. When an attempt is made to split such slate into thinner slabs, it breaks across the cleavage into irregular, worthless slivers. The resulting fractures are called false cleavage or, if massed, cleavage shear zones.

*Jointing.*—Joints are rock fractures due to various causes. They are smooth-walled, regular, open crevices; or curved openings, especially where crossing from one layer to another; or crevices that have since been filled with mineral matter. In many slate regions they are highly regular in pattern. A group of joints may be approximately parallel, all inclining

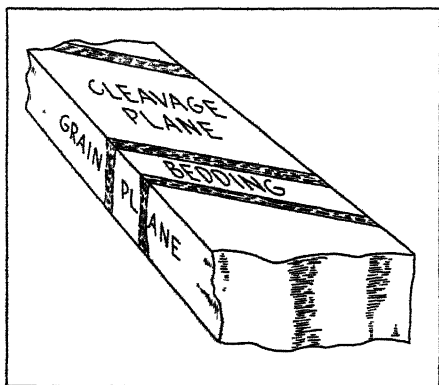


FIG. 3.—STRUCTURAL FEATURES OF BLOCK OF SLATE AS QUARRIED.

or dipping in a single direction. Such parallelism may help in quarrying; on the other hand, numerous joints cutting the cleavage make difficult the winning of large, unfractured blocks from the quarry.

*Bedding.*—Like all rocks originally deposited in layers or strata, slate generally shows traces of stratification or bedding, however much deformed or concealed by subsequent recrystallization. This is important from a practical viewpoint because: (1) as explained, in some slates the successive layers differ in composition; therefore the slate tends to break parallel to the bedding or transverse to the cleavage (see Fig. 2); (2) the color in many slates changes from bed to bed; thus quarried blocks may show both desirable and undesirable colors.

*Conductivity and Resistivity.*—Slate is generally a good heat and electrical insulator. The property of insulating against electricity makes it highly valuable in switchboards and similar places. The chief characteristics of beds serving as electrical insulators are: (1) uniformity of grain

and (2) the absence or relative paucity of the following minerals: magnetite, pyrite, graphite (especially in locally dense areas), and quartz (especially when highly localized).\*

#### DISTRIBUTION OF DEPOSITS

The valuable slate deposits of the world are by no means the only slate deposits known: they are simply those that occur near enough to a considerable market to merit working on a large scale. Therefore virtually all important slate deposits are near densely populated regions.

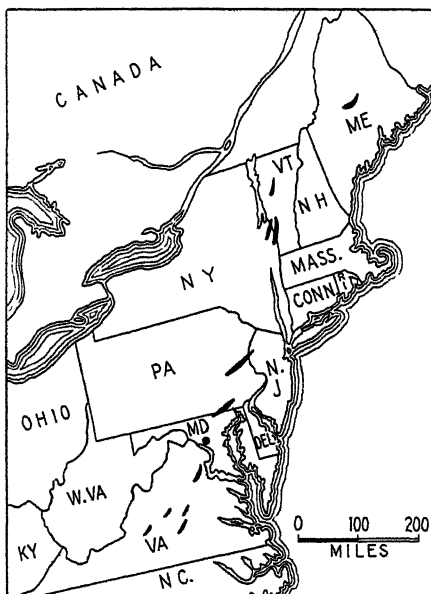


FIG. 4.—GENERALIZED MAP OF CHIEF PRODUCTIVE SLATE BELTS OF THE EASTERN UNITED STATES.

*United States.*—Slate is quarried in small amounts in several localities in the western United States, notably central Colorado,† western Nevada, near Phoenix, Ariz., near Placerville, Calif., and at Provo, Utah.‡

The Eastern States, however, contain the best developed slate deposits. Minor deposits are in the Ouachita Mountains of Arkansas,<sup>22</sup> northwestern Georgia,<sup>25</sup> southeastern Tennessee,§ West Virginia,§ northern Minnesota,§ and New Jersey.§ None of these areas has note-

\* Ref. 3, 101, 184.

† Ref. 8, 70, Plate I.

‡ Ref. 8, 65-70, 119-120.

§ Ref. 8, 115-119, 164-166, 86, 87-88, respectively.

worthy production at present, however. The five outstanding slate regions in the United States (Fig. 4) are described below.

The Maine deposits are near the middle of the state, chiefly around Monson. The slate is of early Palaeozoic age, and is dark bluish gray, with a purplish cast. The nearly parallel position of bedding and cleavage for great distances is surprising.\* Maine has for long been an important producer of electrical slate and has produced minor amounts of roofing and structural slate.

The Vermont-New York slate<sup>7</sup> belt is the second most important in the United States. The slates are Ordovician and Cambrian in age. They are greenish gray, purplish, variegated (i.e., greenish gray and purplish, mixed), and deep brick red. Some of the green slates are "unfading," while others ("sea green") become less green and more gray upon prolonged exposure. This belt usually stands second in the production of roofing and structural slate and leads in the production of electrical slate. Its colored slates, the only domestic colored slates available, are especially desired for roofing and for floor tile and flagging.

The slate belt of eastern Pennsylvania is the most important in this country.† It centers around Bangor, Pen Argyl, and Slatington. Much of the slate is banded in various shades of gray, the dark gray bands being narrower and characterized by a higher carbon content. The gray color makes this slate excellent for blackboards, bulletin boards and the small school slates. The district furnishes the greater part of the nation's roofing slate and about 90 per cent of its structural slate.

The Peach Bottom district‡ produces minor amounts of roofing slate and some structural slate. It extends northeastward across the line separating Pennsylvania and Maryland; centers of production are Cardiff and Delta. The slate is assigned to the pre-Cambrian,<sup>13</sup> and is closely folded; structural details are not clear. It is an excellent roofing slate, lustrous and of dark blue-gray color. An important product in recent years has been slate granules.

The Virginia slate districts may be grouped into three belts, the eastern being of Ordovician, the central and western belts of Cambrian age.§ They have been described by Watson<sup>29</sup> and Dale.|| For the most part, the slates are dark gray with faintly greenish or brownish tints, and highly lustrous, but some dark greenish gray and variegated slate has also been quarried. Virginia slate is used chiefly for roofing and granules.

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\* Ref. 8, 74-83.

† Ref. 2; ref. 3, 127-359.

‡ Ref. 17; ref. 3, 359-390.

§ Ref. 8, 147.

|| Ref. 8, 146-164.



*Canada.*—Production of slate in Canada is not of large volume. In Madoc township, southern Ontario, a good blue-gray slate is known.\* In New Brunswick and Nova Scotia also slate of commercial grade occurs, but the only deposits worked are those in Halifax and Hants Counties, Nova Scotia.† The leading slate districts in Canada are in eastern Quebec,‡ extending from Megantic County southwest to Brome County; these are of Cambrian age and are chiefly blue gray, though some are a deep maroon or a light green color. Though operations began as early as 1854, production is virtually at a standstill in Quebec. Slate is also reported from the western provinces but is not of economic importance at present.§

*Europe.*||—The leading slate-producing countries of Europe, as judged by recent production figures, are Great Britain, France, Belgium and Germany. Slate is produced also in noteworthy amounts in Czechoslovakia, Italy, Norway, Portugal, Spain and Switzerland.

The two outstanding slate districts in the British Isles are Wales and Cornwall. The Welsh quarries and mines are chiefly in Carnarvonshire and Merionethshire; they are opened in Cambrian and Ordovician rocks. Slates of the former age are green, purple and red in color; of the latter age, generally gray or black. Near Dalabole in Cornwall, high-grade slates of Devonian age and bluish gray color are produced. This slate is excellent, but the industry is smaller than in Wales. Slate is also quarried in Scotland, in the Lake district of northern England, and in Ireland.

In France the chief producing centers are along the Meuse River on the north central boundary near Belgium (Department of the Ardennes), and in west central France near the Loire River, in the Department of Maine-et-Loire. Typically the Maine-et-Loire slates are tough, blue-gray in color, and of Ordovician age. In the Ardennes district, they are of Cambrian age, and are purplish, red, green, grayish green and black. Some are especially rich in magnetite.

In Germany the principal slate production in recent years has been in Thuringia and Prussia, with small amounts from Bavaria. Chief producing centers are Bundenbach, Kirn and Caub, all in extreme eastern Germany; Lossnitz in Saxony; Nuttlar in Westphalia; and lesser centers in Thuringia. Most of the slate is blue-gray and of Devonian age. Some is a clay-slate, not wholly metamorphosed into true slate.

The Czechoslovakian slates are greenish and bluish gray and are largely Devonian in age, though in part Mississippian. The Norwegian

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\* Ref. 21, pt. I (1912) 347.

† Ref. 21, pt. II (1914) 199–201.

‡ Ref. 21, pt. III (1914) 235–252.

§ Ref. 21, pt. IV (1916) 275.

|| For a condensed description, the source of much of the information here, see Howe.<sup>12</sup>

TABLE 1.—*World Production of All Types of Slate*<sup>a</sup>  
METRIC TONS UNLESS OTHERWISE INDICATED

	1928	1929	1930	1931	1932	1933	1934	1935
Australia.	2,053	2,055	943	312	229	713	7,201	31,434
Belgium (pieces)	c	32,590,000	27,470,000	22,570,000	15,010,000	13,810,000	16,520,000	c
Canada.	nil	nil	136	227	227	227	.	c
France.	276,490	261,011	260,800	245,530	195,900	146,550	154,900	156,900
Germany.	123,082	82,666	42,151	40,356	51,622	53,231	100,934	90,996
Great Britain...	305,055	305,678	264,705	246,691	256,899	276,888	295,103	307,752
India, British.	9,297	55,028	8,641	8,839	8,483	11,559	10,341	c
Italy....	18,650	19,900	c	c	c	c	c	c
Japan.....	\$224,985	\$192,837	c	c	c	c	c	c
Spain..	c	11,118	12,232	55,611	64,132	c	c	c
Sweden..	1,185	2,710	2,723	1,965	2,699	836	2,577	2,816
South Africa.	\$ 38,640	\$93,600	\$85,050	\$81,550	\$76,775	\$28,259	305	439
United States <sup>b</sup> .	586,377	607,980	420,580	334,230	257,862	227,888	211,087	232,508
U. S. S. R. (slabs)	c	c	c	c	c	c	102,000,000	164,000,000

<sup>a</sup> Slightly amended from Mineral Industry during 1934 (G. A. Roush editor) 535. New York, 1935. McGraw-Hill Book Co.

<sup>b</sup> Amount sold.

<sup>c</sup> Figures not available.

slates are partly green, partly gray. The slates of Portugal are largely dark gray. Coreglia and Rapallo are important slate-producing centers in Italy.

*Other Regions.*—Slate deposits of more than local importance occur in Japan, British India, South Africa and South Australia.

#### POLITICAL AND COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

Slate is widespread, almost all countries containing at least some deposits, usually in quantities far exceeding domestic needs; therefore political and commercial control of a given slate deposit rests with the country in which it is found. The only exception known to the writer is a large deposit in Portugal said to be commercially controlled by British capital.

There are virtually no estimates of reserves available even for the most closely studied districts.\* The quarrying or mining of slate in any given district ceases not because the reserves are used up but because of a shift in demand.

Because slate deposits are so widespread, production usually centers in localities favored by factors other than the mere occurrence of workable slate, therefore the discussion of all slate deposits is largely a description of the centers of production.

Norway and Portugal are the only important producers omitted from Table 1 for lack of data.

Like other structural materials, slate finds its greatest demand in regions of dense population and high industrial development. Hence the United States, Great Britain, Germany and France are conspicuous consuming as well as producing countries. Great Britain and the United States are the leading consumers; making reasonable assumptions, the value of the slate used in 1933 was £1,533,467 for Great Britain and \$2,713,814 for the United States. Important consuming countries that are themselves significant producers are the Netherlands and Canada.

#### PROSPECTING AND EXPLORATION

The fundamental facts underlying intelligent prospecting have been summarized by several authors both for building stones in general† and for slate in particular.‡

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\* Ref. 3, 5.

† For example: Ries;<sup>24</sup> Merrill, ref. 16, 447–488 and Stiny.<sup>25</sup>

‡ Eckel, ref. 9, 95–126; Dale, ref. 8, 167–181; Behre, ref. 3, 68–73 and Bowles, ref. 5, 232–235.

*Geologic Factors and Their Applications.*—An ideal slate quarry should have but little overburden. It should yield only a moderate amount of water. The slate itself should include no conspicuously siliceous or carbonaceous beds and no small masses especially rich in carbon or other specific minerals (such as magnetite) irregularly distributed; in short, the beds should be uniform throughout their sequence. The strata should stand nearly vertical and have a constant dip or inclination. The slaty cleavage should dip at a low angle to the bedding; but ideally the cleavage should dip at a low angle to the horizontal, so as to facilitate quarrying; these two conditions may be mutually exclusive, in which case steep cleavage is not prohibitive. Faults and “false cleavage” or wrinkling of cleavage should be absent. Ideally the beds should trend (technically,

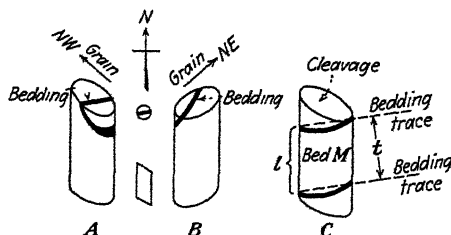


FIG. 5.—ILLUSTRATION OF USE OF DRILL CORES.

A and B, orientation of cores. When regional grain and cleavage trends are known, core is turned so that grain in core is parallel to that in region; thus, if regional grain trends to northwest and regional cleavage (represented by top of core) is to southeast, A is the correct, B the incorrect orientation of core. Notice effect of orientation on dip of beds. Small figures are profile and top views of core. C, determination of correct thickness ( $t$ ) of bed M, as contrasted with distance from top to bottom ( $l$ ) when measured parallel to length of core. Notice that  $t$  is always less than  $l$ .

“strike”) at right angles to the grain and parallel with the cleavage. The joints should be regular and preferably parallel in strike with the strike of the beds; ideally also the joints should dip so as to form an angle of  $90^\circ$  with the dip of the cleavage.

Prospecting aims at finding a site where the relations mentioned occur to as great a degree as possible. A full discussion of these matters has been presented elsewhere.<sup>1</sup>

*Exploration by Means of Surface or Near-surface Data.*—If the slate strata outcrop, the above considerations, coupled with the necessary slate testing and weighing of the commercial factors, will tell whether quarrying is justified. If there is an overburden of soil, gravel or weathered slate, trenches may be dug, directed at right angles to the layers to expose as many strata as possible.

*Core Drilling.*—In the United States, core drilling has been used with some success. Shot and diamond drilling are both employed. The former is less costly per foot drilled but the latter can more easily be

directed at an angle to the vertical, an important factor. Attention to grain, cleavage and bedding may combine to make possible orientation of the core in the correct direction, even if it happens to be turned in removal from the hole (Fig. 5).

### PRODUCTION METHODS

Production methods have been well discussed in several recent publications.\* What follows is a brief treatment only.

*Methods for Breaking Rock.*—For various reasons slate must be quarried more carefully than much other rock. Blasting, because it shatters the rock, has been progressively supplanted by the other methods. For blasting, black powder is preferred.

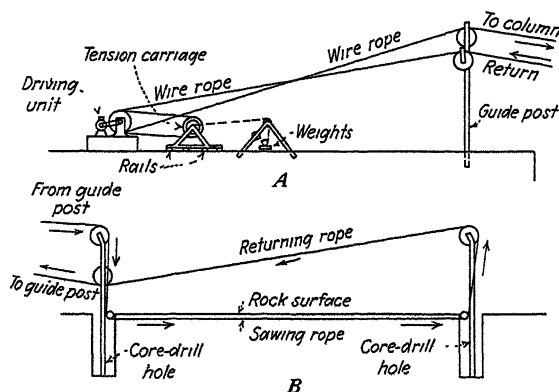


FIG. 6.—DIAGRAM OF WIRE SAW, MODIFIED, AFTER BOWLES.<sup>5</sup>  
A, driving end; B, cutting end.

Drilling is a necessary preliminary step in most quarrying. Hand drilling has been largely supplanted by power drilling. When possible, the less valuable beds are drilled, to reduce waste. Rates of drilling vary; about 15 ft. per hour is an average figure. In wedging parallel with the cleavage metal wedges are forced into the slate by blows with a sledge hammer. Preliminary drilling may be necessary. In quarries in the United States, track channelers are favored, especially in resilient, tough slate. Obviously they can best be used where the cleavage dip of the slate is nearly horizontal.

In Europe the wire saw has been used for some time and since 1928 it has increasingly found favor in America. The "saw" is a twisted steel cable running as an endless belt. Sand and water fed upon the wire serve as the abrasive. The wire is driven by passing over one or two sheave wheels and over a tension device, which keeps it taut. Depth is acquired

\* Ref. 6, 23-123; ref. 26; ref. 3, 84-106.

by two holes drilled into the rock by a 36-in. core drill (Fig. 6). Into these holes standards are placed along which the return wheels are progressively lowered. The method eliminates shattering and reduces waste.\*

*Removal of Blocks.*—Typically the blocks are set free at the quarry walls by channeling, drilling and blasting, drilling and wedging, or sawing with the wire saw. The overburden having been removed, there remains only a single surface, that below, which is yet to be broken free before the block can be hoisted. This freeing is generally done by wedging along the cleavage surface. Chains are attached to the block and it is hoisted out of the quarry. Maximum weights of blocks handled usually do not exceed two tons in American quarries. Hoisting machinery resembles that used in quarrying other kinds of stone. From the landing the slate is transported to the "factory" where it is to be fabricated.

*Slate Mining.*—Where the desired slate beds are thin or the overburden great, handling of much waste is obviated by underground mining. This is done in Wales,† in Germany,<sup>26</sup> and in Pennsylvania.‡ The methods used do not differ greatly from other mining. Where beds are thin and horizontal, pillars are left to support the roof. Where beds are thin but nearly vertical (as locally in Germany), the openings are filled with waste rock for support.

*Effect of Geologic Features on Quarry Methods.*—Quarry methods vary with differing geologic features. Thus, where the cleavage dips steeply, wedging cannot readily be used for lifting blocks from the quarry floor. Where the beds stand vertical, certain beds are selectively quarried; if they are nearly horizontal the proportion of waste in quarrying is likely to be great in gaining access to deeper, more valuable beds.

*Waste Disposal.*—The proportion of waste has been variously estimated at 60 to 85 per cent of the slate quarried. Hence much research has been aimed at both avoidance and utilization of waste.

#### PREPARATION FOR MARKET

Slate requires a high finish. The relatively high costs of slate per unit is due largely to this need for finishing before marketing. Blocks of slate are reduced to workable sizes, with iron circular saws 2 to 4 ft. in diameter and  $\frac{3}{8}$  in. thick, turning about 5 r.p.m. No abrasive is used. Later, large slabs are accurately cut to specified dimensions by rapidly rotating, thin carborundum wheels.

Slate blocks are split by hand parallel to the cleavage. Mechanical

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\* Ref. 5, 255-260. † Ref. 5, 394-375.

‡ Ref. 3, 25, 313, 317, 318-319.

splitting devices have not proved satisfactory. Desirable large blocks are carefully split for blackboards, and small blocks for roofing or school slates. The tools used are thin-bladed chisels or wedges, hand driven. In American practice, a block to be finished for structural uses is planed by means of a straight-bladed knife, mechanically scraped over the cleavage surface of the slate. For finishing slabs for blackboards or for certain structural purposes, the cleavage surface is smoothed with sand on a rotating iron rubbing disk; or by a mechanically moving abrading head; or by hand honing. Slate may be drilled for screw holes and the pieces fitted together into such equipment as tubs, basins and switchboards.

Much slab slate is surfaced either with oil, varnish or black enamel. In England, roofing slate surfaces are being stained with colloidal oxides of iron and manganese. In the United States the surface of structural slate may be "marbleized"—covered with enamel, patterned, for example, in imitation of marble.

#### TESTS AND SPECIFICATIONS

*Tests.*—Numerous publications deal with the testing of building stones in general,\* and of slate in particular.† Hirschwald's discussion is especially recommended as a source for comparisons between slates.‡ Systematic testing methods have also been developed lately under the auspices of the American Society for Testing Materials. What follows presents the subject of slate testing in the briefest possible form; details are given in footnote references.

Properties of slate of conspicuous commercial importance are: (1) cleavability, (2) color and its constancy, (3) density, (4) porosity and absorption, (5) hardness, (6) toughness, (7) transverse and crushing strength, (8) elasticity, (9) electrical resistance, and (10) corrodibility. The quarryman is primarily interested in Nos. 1, 2, 5, 6 and 8. The consumer of roofing slate is chiefly interested in Nos. 2 and 6; for structural uses, Nos. 2, 4, 5, 6, 7, 8 and 10 are of greatest interest; and for electrical purposes No. 9 is paramount. Tests developed for the properties numbered 1 and 2 are largely empirical, though tests for color and color constancy are being devised by the U. S. Bureau of Standards. Specific gravity (or density) is most accurately measured by weighing and obtaining the volume with a volumometer§ or by means of a Le Chatelier flask.<sup>12a</sup>

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\* For good summaries, see the following: Merrill, ref. 16, 447-488; Howe, ref. 12, 362-411; Hirschwald, ref. 11, 53-123; Stiny.<sup>28</sup>

† Dale, ref. 8, 171-181; Behre, ref. 3, 73-81, 101.

‡ Ref. 11, 592-645.

§ Ref. 11, 107-109.

Hardness is best tested by means of a sclerometer\* or Deval abrasion machine.† Toughness is tested with a plunger which drops from a given height.‡ Tests for crushing strength,§ transverse strength,§ and elasticity;§ porosity and absorption are similar to those applied to other structural materials. Of course, the vector properties of slate differ greatly; thus, elasticity and crushing strength are great at right angles to cleavage. Extremes of heat or cold or a large amount of absorbed moisture reduce elasticity, transverse strength and crushing strength.

Laboratory and field devices for measuring electrical resistance<sup>10,19,27</sup> consist of a current source and electrodes placed on opposite sides of the slab to be tested, the current being read by ammeter or voltmeter.

If structural slate is subjected over sufficiently long periods to highly corroding vapors or solutions, it may suffer color changes or partial disintegration. The most injurious agents are sulphurous, sulphuric, sulphuric-hydrochloric (mixed), hydrochloric, nitric and acetic acids, and sodium and ammonium hydroxides.<sup>15,20</sup>

*Specifications.*—Roofing slate is sold in the United States by the "square," the slate necessary to cover 100 sq. ft. with a specified overlap—as a rule, 3 in. A similar unit, the mille, is used in Great Britain. The standard thickness is  $\frac{3}{16}$  in., but greater thicknesses and irregular cleavage are sometimes desired for an "antique" appearance. Standard sizes for roofing slate in the United States range from 10 by 6 to 24 by 14 in. The order generally specifies color, and whether the entire slate must be free from dark beds ("clear") or have such dark beds beneath the overlap ("semi-clear") or exposed to view ("ribbon").

Millstock may not be too heavily "ribboned." The finish is specified—e.g., "sawed" or "split," "planed" or "honed." Dimensions and fittings are described.

Blackboards and bulletin boards, which are sold by the square foot, must be free from cracks, conspicuous beds or other structural blemishes, and are preferably plane, but may be gently curved. The color must be dark gray or black. School slates may be framed before shipping. They must be smooth and dark. Dimensions are specified.

Electrical slate is sold by the square foot. It must be uniformly a poor conductor. Conductivity must be tested. The specifications give sizes.

For crushed slate, size is the prime consideration.

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\* Ref. 11, 83–88.

† Ref. 19, 22.

‡ Amer. Soc. Test. Mat. Tentative Standards for 1924 (1925) 924–925.

§ Ref. 11, 60–61; ref. 12a, 502–503; ref. 8, 175–176; Amer. Soc. Test. Mat. Tentative Standards for 1929 (1930) 547–550.



## USES, MARKETING AND PRICES

*Uses.*—The following list briefly indicates the uses of slate:

1. Roofing:
  - Standard roofing slate.
  - Slabs for "antique" roofs.
2. Millstock:
  - "Wall board" for insulation.
  - Baseboard.
  - Stair treads and risers.
  - Garden walks and flooring.
  - Mantels.
  - Sinks, dripboards, etc.
  - Shower and toilet stalls.
  - Billiard table tops.
  - Grave vaults and covers.
  - Fence posts.
3. Blackboards:
  - Blackboards.
  - Bulletin boards.
4. School slates:
  - Framed or unframed tablets.
  - Slate pencils.
5. Marbleized slate as a decorative stone.
6. Granules:
  - Banded briquettes.
  - Surfacing flexible shingles.
  - Surfacing roll roofing.
7. Pulverized slate:
  - Paint filler.
  - Linoleum filler.
  - Rubber filler.
  - Fuse covers.
  - Substitute for fuller's earth.
  - Pigment (if red or green).
  - Abrasive soaps and polishes.

*Marketing.*—Bowles<sup>4</sup> points out that freight rates largely fix the market range of slate products; thus, in the United States the major part of the sales is confined to the area east of the Ohio drainage and thus near the quarries. Marketing centers are eastern Pennsylvania, New York City, Boston, Vermont, Richmond and Norfolk. Structural and electrical slate may travel west to Chicago and St. Louis.

International trade is chiefly to and from coastal cities—Italian slate enters the eastern United States, and British slate the coastal cities of Holland.

As building standards continue to rise in westerly United States and Canada, probably the frontier of slate sales will be pushed westward. A large expansion of sales cannot be looked for in the near future, however, and slate is already under severe competition from substitutes.

TABLE 2.—*Approximate Sales Values of Slate in the United States*

Material	Value per Unit in 1934	
	Unit	Value
Roofing slate. . . . .	Square	\$7.54
Electrical slate. . . . .	Square foot	0.73
Blackboards. . . . .	Square foot	0.23
School slate. . . . .	100 pieces	0.91
Structural slate. . . . .	Square foot	0.30
Flagstones. . . . .	Square foot	0.07
Granules and slate "flour" . . . . .	Short ton	6.42

*Prices.*—Quarrying fluctuates concurrently with changes in prices. Previous to the World War the prices rose steadily. During the war they remained fairly constant, though production suffered through loss of labor. After the war came a distinct decline, culminating in 1929–1932, from which the slate industry, both abroad and at home, is only now slowly recovering.

As prices vary greatly with the specific product and with time, few general statements can be made. Table 2 shows approximate sales values per unit in the United States.

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## CHAPTER XL

### SODIUM CARBONATE AND SODIUM SULPHATE

By ROGER C. WELLS,\* MEMBER A.I.M.E.

SODA ash and salt cake are two of the basic materials of the modern chemical industries and are used in large amounts. The former is the anhydrous carbonate,  $\text{Na}_2\text{CO}_3$ , and the latter the sulphate,  $\text{Na}_2\text{SO}_4$ , of soda. The name "soda" is employed to designate either the carbonate or the oxide,  $\text{Na}_2\text{O}$ , of sodium the metal. The production of these compounds is often referred to as the "alkali industry."

Natural salts constitute only a small percentage of the total production of these compounds in this country. The bulk of the production comes from chemical works that use other salts, largely sodium chloride, as raw material. Imported sodium sulphate is also made from waste magnesium sulphate in the German potash industry. At a few places in the Western States, however, both sodium carbonate and sodium sulphate occur naturally in considerable quantity. The present discussion is restricted wholly to these natural salts.

Sodium carbonate is found in solution in alkali lakes, and as the mineral thermonatrite<sup>4</sup> ( $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ ); nahcolite ( $\text{NaHCO}_3$ ); trona ( $\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot 2\text{H}_2\text{O}$ ); and in different double carbonates such as gaylussite ( $\text{Na}_2\text{CO}_3 \cdot \text{CaCO}_3 \cdot 5\text{H}_2\text{O}$ ) and others. Sodium sulphate is also found dissolved in salt lakes and as the mineral thenardite ( $\text{Na}_2\text{SO}_4$ ); Glauber's salt, or mirabilite ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ); and in double salts such as apthitalite [ $(\text{Na}, \text{K})_2\text{SO}_4$ ]. Both salts are combined in the mineral burkeite ( $\text{Na}_2\text{CO}_3 \cdot \text{Na}_2\text{SO}_4$ ), which occurs at Searles Lake but not in sufficient quantities to mine directly. They are also present in hanksite ( $9\text{Na}_2\text{SO}_4 \cdot 2\text{Na}_2\text{CO}_3 \cdot \text{KCl}$ ). The chief sources of both salts are the brines of the alkali lakes, especially Owens Lake and Searles Lake, reefs of trona, and deposits of thenardite and Glauber's salt in the arid regions of the Western States.<sup>1, 5, 10</sup>

The sodium carbonate minerals dissolve in water to give an alkaline reaction. Solutions of the sodium sulphate minerals are neutral. The minerals differ widely in solubility, according to their composition, the temperature, and presence of other salts in solution. The solubilities

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<sup>4</sup> References are at the end of the chapter.

of the several salts are important in explaining the origin of the minerals and in the chemical engineering methods required to separate and purify the different salts. They are best represented by phase-rule diagrams such as those given by Wells,<sup>14</sup> Teeple,<sup>12</sup> Cole,<sup>3</sup> and others. The most characteristic feature of both salts is that they form hydrated salts with 10 molecules of water of crystallization at low temperatures, but a monohydrated carbonate above 35.4° C. and an anhydrous sulphate above 31.4° C.

#### ORIGIN OF THE MINERALS

A number of different theories have been proposed to account for the origin of these particular saline minerals, some of them fairly obvious, others more difficult to prove with the information available, and considerable geochemical reasoning is required to cover the whole story. Although sodium is the sixth element in order of abundance in the earth's crust, most of it is in the igneous rocks, from which it is slowly leached to pass into surface waters and thence to the ocean. Where rainfall is restricted by mountain ranges, however, leached salts may accumulate in local basins of small or great extent by evaporation of the scanty rainfall. If the attack on the rocks were with pure water only, the resulting solutions would generally be strongly alkaline,<sup>11</sup> but this alkalinity is reduced by carbon dioxide absorbed from the atmosphere, by sulphuric acid produced by the oxidation of sulphides, and by other acids contributed by volcanic action. The resulting products include carbonates, bicarbonates, sulphates, chlorides, fluorides and borates. The mere evaporation of the solutions leached from igneous rocks would therefore yield some sodium carbonate and sodium sulphate directly, principally sodium carbonate.

The average igneous rock does not contain enough sulphur, however, to form much sulphate, relative to the possible carbonate, so that an additional supply seems to be required to explain the origin of sodium sulphate deposits. Aside from volcanic sources and hot springs, this supply is apparently furnished by the widespread presence of gypsum deposits in the arid regions and in sedimentary rocks. Where gypsum is dissolved and the solution mixed with a sodium carbonate solution the combination formed on a second evaporation will be calcium carbonate and sodium sulphate. Cole,<sup>3</sup> though tracing the sulphate radical to gypsum, ascribes the origin of the sodium to base-exchange silicates, such as bentonite. These, however, are only a step removed from original igneous rock, therefore it seems reasonable to ascribe the origin of sodium carbonate to igneous rocks directly, that of sodium sulphate to the same source in part, as well as indirectly through repeated evaporation involving gypsum as an intermediate agent. The typical sodium sulphate

ponds of Wyoming seem to be the product of repeated evaporations, as they contain little carbonate. The carbonate lakes of Oregon, on the other hand, are little removed from the parent volcanic rock.

All deposits of these salts are of recent age, geologically. The salts are moderately soluble, though not as soluble as the salts found in bitterns.

#### DISTRIBUTION OF DEPOSITS

Natural deposits of sodium carbonate and sodium sulphate in the United States are confined to the Western States—Arizona, California, Nevada, New Mexico, North Dakota, Oregon, Texas, Utah, Washington and Wyoming. The exact locations have been given so many times that they need not be repeated in detail here.<sup>13</sup> Moreover, the mere existence of many occurrences is of little significance, as only the largest deposits worked by the best chemical engineering methods seem capable of steady production. Searles Lake has proved unique. The relative freedom from clay and sedimentary material of the salts in this deposit may be due to the fact that the solutions from which the salts were deposited overflowed from other valleys. In 1914 the United States Geological Survey sank several test wells in the Black Rock Desert, north of Trego, Nev., primarily for potash; the search for that mineral was not successful, but the valley was found to be filled for the most part with clays and silts. In the main well, which went to a depth of 1465 ft., three small flows of alkaline water were encountered at 5 ft., 503 ft. and 1389 ft., respectively.

TABLE 1.—*Analyses of Brines*

Constituent	Composition, Per Cent		Constituent	Composition, Per Cent	
	Searles Lake Brine <sup>a</sup>	Salts from Composite Sample of Muds from Black Rock Desert, Nevada <sup>b</sup>		Searles Lake Brine <sup>a</sup>	Salts from Composite Sample of Muds from Black Rock Desert, Nevada <sup>b</sup>
SiO <sub>2</sub> ...		0.40	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> . . . .	0.43	0.67
NaCl....	16.33	21.51	Na <sub>2</sub> HPO <sub>4</sub> ...	0.14	
Na <sub>2</sub> SO <sub>4</sub> ..	6.98	4.62	NaBr.... . . . .	0.11	
KCl....	4.69	0.56	LiCl..... . . . .	0.02	
Na <sub>2</sub> CO <sub>3</sub> ...	3.46	2.24	Na <sub>2</sub> S..... . . . .	0.02	
NaHCO <sub>3</sub> .	0.77		As <sub>2</sub> O <sub>3</sub> ..... . . . .	0.02	
Na <sub>2</sub> B <sub>2</sub> O <sub>4</sub> .	1.39		H <sub>2</sub> O... . . . .	65.64	70.00
				100.00	100.00

<sup>a</sup> Analysis by R. C. Wells, with minor constituents from Teepie.

<sup>b</sup> Analysis by R. K. Bailey, computed for a saturated brine.

The soluble salts were 10 per cent, 2.6 per cent and 11 per cent, respectively, largely sodium chloride. The first and third waters also showed small percentages of sodium carbonate and sodium sulphate, whereas the second had only a little sodium bicarbonate in addition to salt. The clay samples recovered from the main well showed from 2 to 9 per cent of soluble salts, again largely sodium chloride, but with some sulphate and carbonate. A typical analysis of the salts extracted from the muds is given in Table 1. Deposits of soluble salts in the desert basins are protected by covers of clay and mud in many places, so that more dilute waters may be found either above or below them. Sodium sulphate has been produced at Rhodes Marsh, 8 miles north of Mina, Nevada.

Deposits of impure gaylussite containing about two million tons have been reported near the surface in Railroad Valley, Nevada, T.7N., R.56 E., a foot or more in thickness, covered by a few inches of mud and sodium sulphate. Gaylussite was also found in beds several feet thick at depth of about 800 ft. No attempts have been made to utilize these deposits.

Omak Lake, Okanogan County, Washington, contains a considerable quantity of sodium carbonate, with some sodium sulphate, but in the form of a relatively dilute brine.

Natural soda occurs at several places in Lake County, Oregon, and some was produced in 1916, but the region is far from any railroad. Summer and Abert Lakes contain considerable sodium carbonate in solution, and mud along the shore of Summer Lake also carries from 10 to 15 per cent of sodium carbonate. The dry lake in T.30S., R.22 and 23E., is said to be filled with volcanic ash, brine, and crystals of soda, which are concentrated in several so-called pot holes. The writer has not seen this deposit.

At Green River, Wyoming, a sodium carbonate brine pumped from wells has been worked for soda at different times and a small production was reported for 1935.

The recent new discoveries of sodium sulphate in northwestern North Dakota<sup>7</sup> deserve special mention because of the size of the salt beds. Plans are now under way to work these deposits.

Deposits of sodium carbonate and sulphate are found in Manitoba, Saskatchewan, Alberta and British Columbia, Canada; in Mexico, Russia, Spain, Rumania, Siberia, Armenia, Persia, Hindustan, Tibet, Tartary, China, India, Turkestan, Arabia, Egypt, British East Africa, Union of South Africa, Peru and Chile. In addition to these places where the salts have been used to some extent locally there are many others where the same substances are known to occur in small quantities, either in surface ponds, or too remote from markets to be worked.

## TARIFFS

The production of these salts is regulated by political factors to a limited extent. The United States tariff on soda ash is one-quarter of a cent per pound, though the bicarbonate is on the free list. Anhydrous sodium sulphate nominally carries a duty of \$3 a ton, and Glauber's salt \$1 a ton, but as crude sodium sulphate, or salt cake, is on the free list, the imported material comes in as salt cake.

## PRODUCTION

The production of the two natural salts in the United States from 1925 to 1935 is shown in Fig. 1. World production is not known.

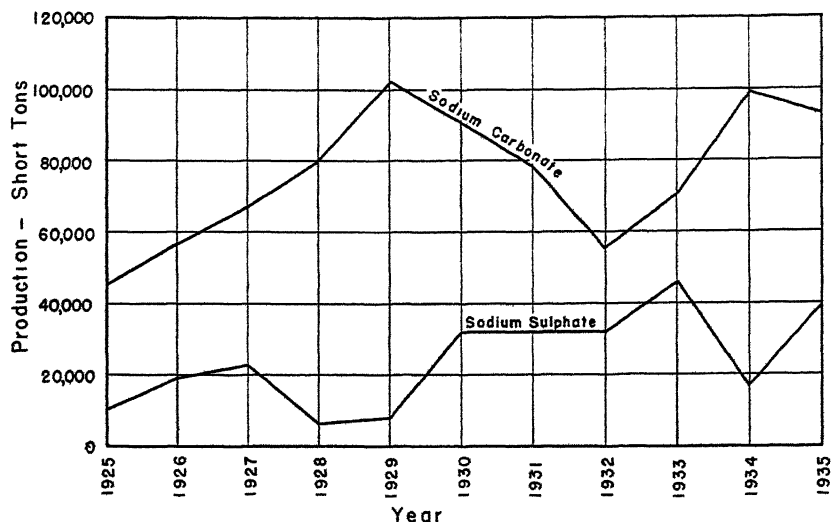


FIG. 1.—PRODUCTION OF NATURAL SODIUM CARBONATE AND SODIUM SULPHATE.  
Compiled by A. T. Coons, of the U. S. Bureau of Mines.

The soda lakes of Egypt were utilized centuries ago, and natural soda in the United States may have been used by the aborigines, but the first recorded notice seems to be that of Rev. Samuel Parker, who, in his journey across the Rocky Mountains in 1835, observed what he called "epsom salts" near Independence Rock, Wyoming. The "natural saleratus" along the Sweetwater River is said to have been known to and used by the Mormon pioneers in 1849. Water high in sodium carbonate was struck at Green River, Wyoming, in 1895, at a depth of 125 ft. Farther west the natural soda at Ragtown, Nev., was utilized



about 1868. Several works were in existence in Nevada in the early seventies, but methods were too crude to be effective.

Operations were begun at Keeler, on Owens Lake, California, by the Inyo Development Co. in 1885 and continued down to 1920. The treatment consisted essentially in evaporation of the water, separation of trona, and calcination of the latter to soda ash. A second and third company followed at Owens Lake in 1912 and 1917, respectively, but the Inyo Development was combined with the California Alkali Co. in 1919, making two companies at Owens Lake at that time. A third company began operations at Owens Lake in 1928. The diversion of the water of Owens River to Los Angeles as the municipal water supply in 1917 allowed Owens Lake to evaporate until it became, like Searles Lake, nothing but a vast bed of different salts with its interstices filled with brine. Since 1932 two companies have produced soda at Owens Lake.

Experiments were begun at Searles Lake about 1905 and the American Trona Corporation was organized in 1913. The only soda made at Searles Lake up to 1927, however, was made from trona. The West End Chemical Co. began producing soda ash in 1928 and has continued to date. The Trona Corporation, now the American Potash and Chemical Co., has produced both sodium carbonate and sodium sulphate since 1934, as well as potassium chloride and borax.

At the present time three of the companies that produce soda in California also produce borax. Efforts to produce sodium carbonate in Oregon and Wyoming have not been very successful.

Sodium sulphate has been produced in Natrona County, Wyoming, since 1917, at least, and in Carbon County since 1930. The deposits are ponds near Natrona, Casper, and about 28 miles north of Rawlins. At present sodium sulphate is also being produced in California, as already mentioned, at Okanogan, Wash., and near Monahans, Ward County, Texas. Operations near McKittrick, Calif., Wabuska, Nev., and Camp Verde, Ariz., have been discontinued.

#### PROSPECTING AND MINING

Prospecting for these salts has not been done in a very systematic way heretofore. Samples collected in the field have generally been sent to the laboratory for analysis or identification by means of the microscope. Hand augers or field rigs have been used for shallow tests but the salt beds are so hard that a sturdy rig is necessary. Core samples are most satisfactory. In drilling for cores the nature of the drilling solution is important. If only water is used the more soluble salts are apt to be dissolved and lost, though the solution may give a clue to their possible presence. A solution initially saturated with the salts involved will permit recovery of the solid salts but it may also impregnate porous

samples with the solutions and lead to confusing results, as the salts are present in small percentages in most of the desert basins.

The mining of sodium carbonate consists merely in pumping the brine either to evaporating basins, as at Owens Lake, or to evaporators in the plant, as at Searles Lake. The composition of the brine of Searles Lake is shown in Table 1. This brine is treated to yield several salts. The process is sketched by Teeple.<sup>12</sup> In outline it consists in evaporating the brine in multiple-effect evaporators in which the salts  $\text{NaCl}$ ,  $\text{Na}_2\text{SO}_4$  and  $\text{Na}_2\text{CO}_3$  separate, the last two as the double salt burkeite,  $\text{Na}_2\text{CO}_3 \cdot \text{Na}_2\text{SO}_4$ . The resulting solution is allowed to settle, then cooled quickly to deposit potassium chloride, and the remaining solution is agitated with air and slightly acidified to deposit borax,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ . Further recrystallization of the first three salts yields the separate products,  $\text{Na}_2\text{CO}_3$  and  $\text{Na}_2\text{SO}_4$ .

Sodium sulphate is mined in many places by blasting out the crude solid salt and in other places by leaching out a warm saturated solution from the crude salt, which is then cooled to precipitate Glauber's salt or evaporated to yield the anhydrous salt. At Casper, Wyo., two ponds are used and the brine is pumped from one pond to the other in order to get at each new crop of solid Glauber's salt. Sodium sulphate has been purified at the Great Salt Lake by dissolving crude Glauber's salt, found under sand along the shore in places and also deposited in winter, and evaporating the solution to deposit anhydrous sodium sulphate. At Wabuska, Nev., the temperature is high enough in the dry season to yield thenardite on solar evaporation. Other ways to prepare the anhydrous salt are summarized by Tyler,<sup>13</sup> Rich,<sup>9</sup> and McDermid.<sup>8</sup> Proposals have been made to make sodium sulphate from polyhalite and in other ways, but none of these processes is being used, so far as known to the writer.

#### SPECIFICATIONS

The specifications generally required on the salts made from natural sources are the same as those for the chemical products, soda ash and soda cake. They are given by Cole,<sup>3</sup> together with methods of analysis, which, however, are the usual analytical methods used, for example, in analyzing brines and natural waters.

#### USES

Sodium carbonate has a great many uses and is one of the basic chemicals of the chemical industry. The anhydrous salt, soda ash, is used largely in the manufacture of glass, soap and other sodium compounds. Caustic soda is made from the carbonate in the larger chemical works and used in making dyes, cleansing agents, and soaps; in refining

petroleum, cleaning metals and in making organic chemicals. However, the proportion of natural soda ash used in making caustic soda is small. The hydrated sodas are used as washing soda, washing powders, detergents, etc. The bicarbonate of soda is used in baking, in fire extinguishers, in making "soda water" and as a chemical.

Sodium sulphate is used in making wood pulp for kraft paper and paper board, and in making glass, in dyeing and for other purposes. In the so-called sulphate process of making wood pulp, only enough sodium sulphate is used to replace losses in a cyclic process. The waste liquors are evaporated with their load of carbonaceous matter and salts and the residue finally heated with the requisite quantity of fresh sodium sulphate. The product is dissolved in water and causticized with lime, yielding a solution of sodium hydroxide and sodium sulphide, which is suitable for

TABLE 2.—*Uses of Sodium Carbonate and Sodium Sulphate*

Uses	Sodium Carbonate, Per Cent	Uses	Sodium Sulphate, Per Cent
Glass	29	Pulp and paper	28
Sodium hydroxide	20	Heavy chemicals	26
Soap, cleansers	16	Rayon and textiles	25
Other sodium compounds	13	Glass and ceramics	12
Dyes	4	Dyes and coal tar	5
Exports	3	Other	4
Varied	15		
	100		100

acting on a fresh batch of long-fibered wood, after the solution is separated from the calcium carbonate sludge formed in causticizing. There are wood-pulp plants in Alabama, Arkansas, Florida, Georgia, Louisiana, Michigan, Mississippi, New Hampshire, New York, Pennsylvania and Wisconsin.

In making glass, the sand, soda ash, sodium sulphate, coke, and other materials, are mixed in the proper proportions and heated in fire-clay pots until the gaseous products escape. Sodium sulphate is used because it is cheaper than soda ash, although it requires a reducing agent such as coke. For making glass, the salt must be purer than for making kraft pulp, 2 per cent being about the limit for impurities.

The hydrous form of the sulphate, Glauber's salt, is used in dyeing and as a medicine, especially for cattle. The estimated percentages of the two main salts used for different purposes are given in Table 2.

#### MARKETS

To supply eastern markets, the natural compounds must meet high freight charges and compete with imports from foreign sources, so that

their consumption is necessarily limited mainly to places near their source. It is true that the supply of chemical salt cake depends somewhat on the extent to which hydrochloric acid continues to be made from salt and sulphuric acid. The manufacture of soda ash from salt was formerly confined chiefly to the northeastern part of the country but recently three new plants have been built along the Gulf Coast. Cheap raw materials are available in this area and the products can be shipped by both land and water to a number of important consuming centers. The raw materials include clam-shell and oyster-shell beds for lime, salt domes for brine and natural gas for fuel. The new plants are those of the Solvay Process Co., a subsidiary of the Allied Chemical and Dye Corporation, at Baton Rouge, La., the Mathieson Alkali Works at Lake Charles, La., and the Southern Alkali Corporation at Corpus Christi, Texas. The latter company is jointly owned by the Columbia Alkali Co., a subsidiary of the Pittsburgh Plate Glass Corporation, and the American Cyanamid Co. It is estimated that the three new plants will have a total annual capacity of more than 350,000 tons of soda ash, compared with a present total capacity in this country of more than 3,000,000 tons. This would appear to leave the field for the natural soda ash to the western part of the country, unless it can be made more cheaply than by the Solvay process, which seems a fair possibility on account of the simultaneous production of four salts.

The price of natural sodium carbonate has fallen from \$20.30 per short ton in 1926 to \$12.60 in 1935, with an average of \$16.60. The price of natural sodium sulphate has been steadier, averaging \$7 per short ton.

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## CHAPTER XLI

### SPECIAL SANDS

By H. RIES,\* MEMBER A.I.M.E.

**S**PECIAL sands are those that are employed for special purposes. They have a limited use, as compared with sands for concrete and plaster. The sands discussed in this chapter are those used for foundry purposes, glass manufacture, filter beds, abrasive purposes, sand-blasting, engine sand, sand for sheet asphalt, and furnace sand. These sands are almost invariably high in silica, consisting chiefly of quartz grains. In some—for instance, glass sands—freedom from ingredients other than silica, such as iron oxide, is important. Certain grades, like filter sand, require rather careful sizing, and in foundry sands the bond, usually clay, is important.

One deposit may sometimes be used to supply several grades or types of sand by appropriate methods of screening and washing. The special sands discussed here are usually obtained from deposits of unconsolidated material, but sands high in silica are derived occasionally from soft sandstones that can be disintegrated easily. Requirements are not the same for all uses, and, while certain properties may be demanded, there are no officially adopted standard tests except for foundry sands.

#### FOUNDRY SANDS

*Definition.*—Foundry sands include those siliceous sands that are used to make the forms for casting metals. If these are employed for making molds, they are known as molding sands; if for the cores that form the hollow spaces in the casting, they are called core sands.

Naturally bonded sands are those containing a variable, though not usually large, amount of clay, which serves as a bond. Synthetic sands, now much used, represent an artificial mixture of sand with impure clay, fire clay or bentonite. Core sands may be bonded with oil, cereal binders, resin, pitch, etc. The foundrymen apply the term “sharp sand” to one that is free from bond.

*Properties.*—The important properties are fineness, bonding strength, permeability, sintering point and durability—in other words, physical properties. More attention has probably been given to research on the

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properties of foundry sands, and the development of standard methods of testing them, than to any other special sands.<sup>12,14,17</sup>

*Character of Grains.*—The grains of a foundry sand range from about 3 mm. in diameter to particles small enough to be called clay. The sand grains are usually subangular to angular, rounded grains being rare and occurring mostly in sizes coarser than 60 mesh.<sup>16</sup> The surface of the grains

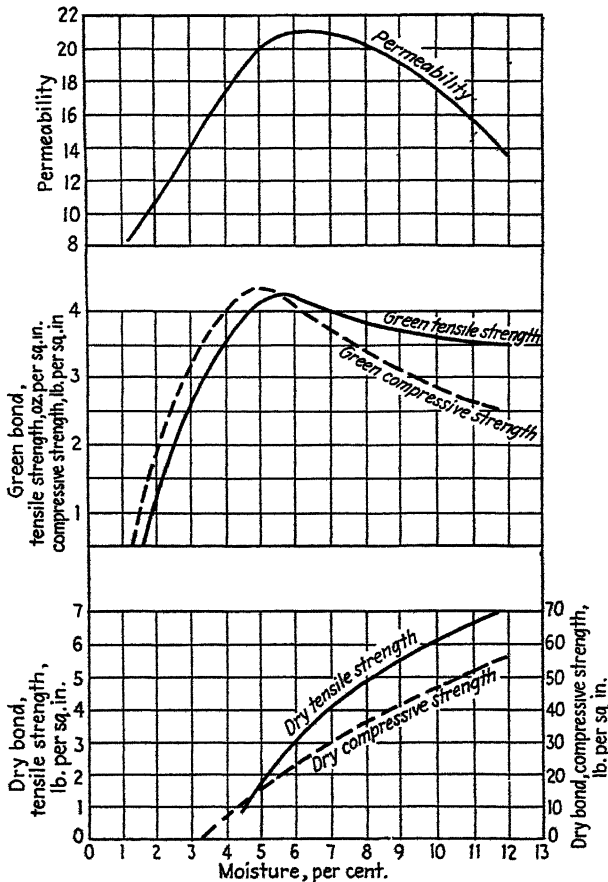


FIG. 1.—PERMEABILITY AND BONDING STRENGTH OF A MALLEABLE IRON SAND AT DIFFERENT MOISTURE CONTENTS. (AFTER A. A. GRUBB.)

may be smooth or rough, clean or stained. Some sands contain compound grains.

*Fineness.*—This term refers to the size of the grains. The American Foundrymen's Association classes as "clay" material finer than 20 microns, while the larger sizes are referred to as "grain."<sup>17</sup> Sands in which large grains predominate are called coarse, while those with a predominance of small grains are called fine. The fineness of a sand is

<sup>12</sup> References are at the end of the chapter.



expressed by the "grain-fineness number," which represents approximately the number of mesh per inch of the sieve that would just pass the sample if its grains were of a uniform size.<sup>17</sup> The American Foundrymen's Association divides sands into different classes, based on the grain-fineness number and the percentage of clay.<sup>17</sup> The fineness of the sand exerts an influence on the permeability and strength, as well as the smoothness of the casting. Table 1 shows the variation in texture of a range of sands, and their other properties for the corresponding grades.

*Bonding Strength.*—The bonding strength of a sand may be expressed in terms of compression, tension or shear. The compressive strength is tested probably oftener than the other two. The strength may be tested either when the sand is moist (green) or after it is dried. Other things being equal, the green strength depends largely on the amount of clay present, as well as on the quality of the latter. The amount of moisture, however, also exerts an influence, as it is found that for every sand there is an optimum water content that gives the maximum strength, and that if the moisture content is increased in one direction, or decreased in the other, the strength diminishes. Dry strength increases with the moisture content of the moist sand (Fig. 1).

*Permeability.*—Permeability is the physical property of the sand that permits the passage of gases. Upon this property of permeability the venting qualities of sand molds and cores depend. The permeability is determined by measuring the rate of flow of air through a standard specimen of sand under a given pressure. Fine sands have a low permeability, while coarse ones are more permeable (Table 1). Clay decreases the permeability. Variation in moisture content affects the permeability in a manner similar to its effect on strength (Fig. 1).

*Sintering Point.*—This represents the point at which fusion of the sand begins. It is important, as it is the temperature at which the sand sinters and "burns on" the casting, thus making it difficult to clean the latter.<sup>17</sup>

TABLE 2.—*Properties of Molding Sands*

Property	Brass Sand			Iron Foundry Sand		
	Light	Medium	Heavy	Large	Medium	Small Casting
Fineness, number.....	220-225	150-200	100-150	25-50	50-100	100-150
Clay, per cent.....	6-15	7-10	7-10	10-15	10-15	10-20
Permeability, per cent. ....	8-12	10-17	17-25	125 up	50-100	20-50
Green, bond tensile, oz.....	4-5	4.3-5.3	4.5-6.0	6-10	4-9	4-9
Compression, lb. per sq. in. ....	3-4.5	3-5	3.5-6.0	6-10	4-9	3-8

*Durability.*—The durability, or life, of a molding sand is its ability to regain most of its green bonding strength after it has been heated by use and is once again mixed with water. Different sands vary in their

durability or life. Table 2 gives the properties of some common types of sand.<sup>12</sup>

*Origin and Mode of Occurrence.*—Most foundry sands are sedimentary deposits of lacustrine or marine origin. They occur in beds of varying thickness, and several different grades may occur in the same deposit. These can be separated in excavating the material. Dune sands are sometimes used as a source of supply for core mixtures. Soft white sandstones are sometimes quarried for producing a high-silica sand.

*Distribution.*—While molding sand is obtained locally by many foundries, there are some important centers of production. Prominent among these is the Hudson Valley north and south of Albany, N. Y. Large quantities are also obtained in the Tertiary formations of New Jersey, some being of very coarse texture, and spoken of in the trade as gravel. Ohio is also an important producer, pits being worked in the northern part of the state near Conneaut, and also in the region around Zanesville. Indiana contributes much as well. The Ottawa district in Illinois supplies much silica sand, while dune sand is obtained from northern Indiana and other localities. Scattered pits of molding sand are worked in a number of other states. Appreciable amounts are obtained in California.

*Political and Commercial Control.*—This factor enters into the foundry-sand business to but a limited degree, as this country is well supplied. Silica sand from Europe has been shipped by water to Seattle, chiefly because none has been developed near at hand. Sand for bronze casting has been obtained from Europe, but the quantity required is limited. On the whole, there is little importation of sand. California sand is shipped to Hawaii for the foundries there, as no local supply has been found.

*Production.*—The output of foundry sand is closely related to the condition of the foundry industry. The production for the past 10 years and price per ton are given in Table 3.

TABLE 3.—*Production of Foundry Sand\**

Year	Short Tons	Value per Ton (f.o.b.)	Year	Short Tons	Value per Ton (f.o.b.)
1926	5,309,166	\$1.06	1931	2,138,305	\$0.99
1927	4,194,975	1.06	1932	1,118,146	0.94
1928	4,781,765	1.06	1933	1,700,000	0.98
1929	6,195,343	1.03	1934	2,240,000	0.95
1930	3,336,855	1.06	1935	2,167,781	1.00

\* Minerals Yearbook, U. S. Bureau of Mines.

*Mining.*—Molding sands are mined by open cut. Steam shovels can be employed where the beds are thick enough, but if thin.

*Preparation for Market.*—High-silica sands such as are employed for cores and synthetic mixtures are usually screened and sometimes also washed. Clay-bonded molding sand may be screened to remove stones, clay lumps and roots, although this treatment is not always satisfactory if the sand is wet or moist. Either stationary or revolving screens are employed. Some plants put the sand through a milling process: (1) to make the product more uniform; (2) for blending two sands, so as to get a product of the desired quality, or (3) to incorporate clay into one that is deficient in bond.

*Tests and Specifications.*—The tests commonly employed are those adopted as standard by the American Foundrymen's Association.<sup>17</sup> Permeability is determined on "green" sand at different moisture contents, or on the sand dried at 110° C. Strength, which may be compression, tensile or shear, carried out on green sand at different moisture contents. The compression and shear tests may also be made on the dried sand. For both permeability and strength, a standard test piece is used. In testing for fineness, the so-called A.F.A. clay is first removed by washing and settling, after which the remaining grain is separated by means of sieves, the numbers used being 6, 12, 20, 30, 50, 70, 100, 140, 200, 270, pan.\* From the figures so obtained the grain fineness number and grain distribution can be calculated.<sup>17</sup> Sintering test is made to determine the temperature at which the grains stick together owing to incipient fusion. There are no standard specifications in the foundry industry, but the consumer sometimes purchases sand according to its grade number, which is not the same at all pits; at other times he may specify that the sand shall show a certain grain fineness number, strength and permeability, with a tolerance allowance if he chooses.

In foundry control the sand heaps are tested daily to see that the properties of the material do not vary too much, and this has served to decrease casting losses greatly. No general statement can be made regarding allowable variation, since it depends on a variety of factors.

*Marketing and Uses.*—Sand is sold by the ton. In the foundry industry it may be used for molds, cores, facing and parting, depending on its properties and fineness.

#### GLASS SAND

This term is employed for sand used in the manufacture of different grades of glass. It may range from high-silica sand, containing but a small percentage of iron oxide, to material with several per cent less silica and as much as one per cent iron oxide. The grains may be round, subangular, or angular. Those from the Ottawa district, Illinois, are often rounded, but most others are angular to subangular. The

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\* This is the series adopted by the American Foundrymen's Association, and the numbers refer to the National Bureau of Standards sieves.

texture should be such that all grains pass a 20-mesh sieve, while there should be little smaller than 100 mesh.

*Occurrence and Distribution.*—Glass sands are found in a number of geologic formations. Those of New Jersey come from the Tertiary, while the Cretaceous of Maryland also affords a supply. Both of these are unconsolidated deposits. The Oriskany sandstone has been worked for glass sand in West Virginia and Pennsylvania and the St. Peter's sandstone (Ordovician) in Illinois and Missouri. Still other supplies come from the Carboniferous in Indiana and Ohio. Glass sand has been produced in a number of states, but West Virginia, Illinois and Pennsylvania have been the three leading producers, with Missouri, New Jersey and Indiana in a group of secondary rank.

TABLE 4.—*Production of Glass Sand*<sup>a</sup>

Year	Short Tons	Value per Ton (F.o.b.)	Year	Short Tons	Value per Ton (F.o.b.)
1926	2,274,218	\$1.59	1931	1,677,882	\$1.66
1927	2,171,693	1.50	1932	1,370,255	1.65
1928	2,310,828	1.49	1933	1,820,000	1.68
1929	2,219,677	1.71	1934	1,923,614	1.73
1930	1,849,101	1.74	1935	2,180,000	1.70

<sup>a</sup> Minerals Yearbook, U. S. Bureau of Mines.

*Mining of Glass Sand.*—The methods employed for working unconsolidated deposits are much the same as those followed for other grades of sand. Steam-shovel mining is possible, but at many plants suction dredges are employed. This is an advantageous method, as the sand has to be washed, and moreover, it undergoes a scrubbing in passing along the pipe line. Stripping should in some instances be carried on in advance of the working, to prevent impure overburden from caving or washing down into the pit where there is danger of this. The consolidated sandstones that are worked are mostly friable and easily crushed. The quarrying methods depend somewhat on whether the beds are steeply dipping or flat. In places, as in eastern Missouri, underground methods are used.

*Preparation of Glass Sand.*<sup>21</sup>—The method of preparation used depends on the character of the crude rock or sand, as well as on the grade of sand desired. All sands have to be washed. With unconsolidated sands, the sand and water may be delivered to revolving screens covered with 12 to 18-mesh wire cloth, the oversize being taken away by drags or conveyers. The sand of desired texture and the clay pass through the screens, and the excess water carries off the clay.

Where sandstone is used, the rock must first be crushed and ground before it goes to the washers. These consist of a cast-iron screw con-

veyer in a sloping box. As the helix conveys the sand upgrade from one box to another, the overflow water carries off the clay. The washed, dried sand is screened to remove oversize sand and foreign material. Vibrating screens are commonly employed.

In New Jersey, according to Weigel,<sup>21</sup> the sand is delivered to the washers through a pipe line from the dredge:

This pipe discharges either to a smaller distributing tank which takes care of the fluctuations in the flow, or may deliver directly to the first screens—revolving screens covered with from 12 to 18-mesh wire cloth. Drags or conveyors take away the oversize. Excess water that passes with the sand through the screens into the tank overflows and carries much of the clay with it. The washers are in effect drag classifiers, with their low ends in the tank below the screen. They usually consist of two flights of chain, each fitted with two parallel lines of steel plate scrapers 3 in. wide and 18 in. long. The sloping floor of the drag box, or angle of the chains, is about 30° from the horizontal.

*Specifications.*—Glass sand should all pass a No. 20 sieve, but there is a difference of opinion as to the allowable amount smaller than 100 mesh. In any event, the latter is probably small, because most of it is

TABLE 5.—*Grading of Glass Sand*<sup>a</sup>

Passing	Retained on	Per Cent
No. 20 sieve . . . . .		100
No. 20 sieve.. . . .	No. 40 sieve	40 to 60
No. 40 sieve... . .	No. 60 sieve	30 to 40
No. 60 sieve. . . . .	No. 100 sieve	10 to 20
No. 100 sieve . . . . .		0 to 5

CUMULATIVE SCREEN ANALYSES OF GLASS SANDS

Mesh Retained on	Sand <sup>b</sup>										
	1	2	3	4	5	6	7	8	9	10	11
14	0	0	0			0	0				
20	0.4	0.6	1.1	0	0	0.1	0.5			0	0
28	1.5	2.9	6.3	0.1	0.6	3.3	3.8	0	0.7	0.1	3.4
35	4.2	13.0	23.0	19.5	17.0	20.3	17.5	1.1	11.6	2.9	30.5
48	12.3	36.8	52.9	60.0	49.7	62.9	56.9	33.6	42.1	23.1	65.0
65	36.7	72.9	84.5	85.7	73.4	90.3	90.9	78.1	74.3	51.3	82.1
100	81.4	95.7	98.6	98.0	87.0	97.5	98.5	94.6	94.6	77.1	92.9
150	97.5	99.2	99.8	99.8	93.6			98.6	99.3	90.6	98.2

<sup>a</sup> Recommended by American Ceramic Society and the National Bureau of Standards.

<sup>b</sup> 1. Williamstown Junction, N. J.

7. Mapleton, Pa.

2. South Vineland, N. J.

8. Berkeley Springs, Va.

3. Washed and tumbled. Locality not given.

9. Weldron, Ill.

4. Ottawa, Ill.

10. Crystal City, Mo.

5. Dike, Nev.

11. Ottawa, Ill.

6. Mapleton, Pa.

removed by washing. One objection to fines is that they are likely to carry an excess of impurities.

The American Ceramic Society and the National Bureau of Standards recommend the grading of glass sand shown in Table 5, but this is not strictly adhered to by manufacturers.

There are no generally accepted standards for the chemical composition of glass sand, although this property is undoubtedly important. Individual purchasers may have definite specifications. A minimum of iron oxide is desirable, because of its coloring effect on the glass. Table 6 gives the specified compositions for several types of glass sand. Chemical analyses of various glass sands are given in Table 7.

#### FILTER SAND

Sand is used by many municipalities to remove sediment and suspended matter, also bacteria, from water.

TABLE 6.—*Specifications for Chemical Composition of Glass Sands*<sup>a</sup>  
PERCENTAGE COMPOSITION OF SANDS OF VARIOUS QUALITIES BASED ON IGNITED SAMPLES

Qualities	SiO <sub>2</sub> Minimum	Al <sub>2</sub> O <sub>3</sub> Maximum	Fe <sub>2</sub> O <sub>3</sub> Maximum	CaO + MgO Maximum
First quality, optical glass.....	99.8	0.1	0.02	0.1
Second quality, flint-glass containers and tableware	98.5	0.5	0.035	0.2
Third quality, flint glass.....	95.0	4.0	0.035	0.5
Fourth quality, sheet glass, rolled and polished plate	98.5	0.5	0.06	0.5
Fifth quality, sheet glass, rolled and polished plate	95.0	4.0	0.06	0.5
Sixth quality, green glass containers and window glass ..	98.0	0.5	0.3	0.5
Seventh quality, green glass.....	95.0	4.0	0.3	0.5
Eighth quality, amber glass containers.....	98.0	0.5	1.0	0.5
Ninth quality, amber.....	95.0	4.0	1.0	0.5

<sup>a</sup> Recommended by American Ceramic Society and National Bureau of Standards.

TABLE 7.—*Chemical Analyses of Glass Sands*<sup>a</sup>

Source	Composition, Per Cent					
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Ignition
Crystal City, Mo.:						
Crude.....	99.405	0.210	0.075	0.072	0.068	0.170
Washed.....	99.78					
Mapleton, Pa.....	99.82	0.12	0.017	Tr.	Tr.	
Hancock, W. Va. ....	99.81	0.17	0.014	0.00	0.00	
Ottawa, Ill. ....	99.607	0.160	0.021	0.050	0.03	0.08
W. Vienna, N. Y.....	98.6	0.17	0.23		Tr.	

<sup>a</sup> From Weigel.<sup>21</sup>

*Properties.*—The essentials of a filter sand are that it shall be of fairly uniform texture, and that the grain size shall be within certain limits. The grains should be of such a character that they will not be disintegrated by the water. Sands high in silica are regarded as the best. Not more than 2 per cent should be soluble in hydrochloric acid. The shape of grain varies from rounded to angular; and, so far as known, these shapes are equally efficient. Flat or elongated grains are undesirable.

*Grading.*—Two terms used to control the average size and uniformity of filter sands are the “effective size” and “uniformity coefficient.”

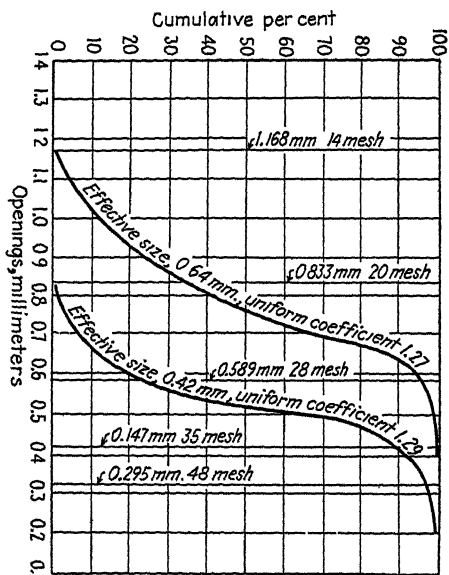


FIG. 2.—CURVES OF SCREEN ANALYSES OF FILTER SANDS. (AFTER WEIGEL.)

The effective size of the sand is that size than which 10 per cent by weight of the sand is finer and 90 per cent is coarser. It is expressed in millimeters, and is determined from a sieve analysis. The uniformity coefficient is the ratio to the effective size of the size than which 60 per cent of the sand is finer. If the grains are of uniform size, the uniformity coefficient will be 1. Fig. 2 gives the cumulative screen analysis of two sands, as well as their effective size and uniformity coefficient.

It is stated that sands with effective sizes of 0.20 to 0.70 mm. are used for filtration, but those most commonly specified have effective sizes from 0.35 to 0.65 mm. The uniformity coefficient varies from 1.25 to 1.80, with 1.55 to 1.60 the average.

*Distribution.*—Filter sand has been produced in a number of states from deposits similar in origin to glass sands. Indeed, the two may be obtained from the same formation. New Jersey is the largest producer,

but much is also obtained from Illinois and Minnesota. A suitable sand can be produced from any good sand bed if it can be properly washed and contains grains of the right size and suitable durability.

*Marketing and Production.*—Owing to the careful preparation required for filter sand, it commands a better market price than ordinary sands. Table 8 gives production and price for the last 10 years. The noticeable

TABLE 8.—*Production of Filter Sand*<sup>a</sup>

Year	Short Tons	Value per Ton (F.o.b.)	Year	Short Tons	Value per Ton (F.o.b.)
1926	79,382	\$2 13	1931	55,319	\$2 17
1927	74,674	2 08	1932	68,035	1 36
1928	113,978	1 99	1933	25,000	1 75
1929	100,081	2 00	1934	35,750	2 39
1930	80,326	2 00	1935	50,000	1 90

<sup>a</sup> Minerals Yearbook, U. S. Bureau of Mines

drop in production since 1929 is probably to be attributed to greater care of the filter beds, the sand being cleaned and used over. It may also be due to the use of granular anthracite, or a thinner sand bed, with gravel underneath.

#### ABRASIVE SAND

Abrasive sand includes all natural sands used for abrasive and grinding purposes. They should be composed of hard grains, and, generally speaking, have a high percentage of silica. They may be used for sawing stone, grinding glass, sandblasting and sandpaper. Those used for different purposes vary somewhat in their character. Artificial sands made by crushing garnet and emery are treated under abrasives.

*Stone-sawing Sand.*—Sand for sawing stone is commonly composed of tough grains, and uniformity of size is desirable, fine material being objectionable. However, unsorted sand is often employed. Weigel<sup>21</sup> states that one large marble company uses a clean, washed river sand with 3.7 per cent retained on 10 mesh, 12.6 per cent on 20 mesh, 83.8 per cent on 48 mesh and 98.6 on 100 mesh. The sand after use can be washed to remove the fines. There are no uniform requirements regarding shape of grain. The sand should be siliceous.

*Glass-grinding Sand.*—This is used for rough grinding of crude-rolled plate glass. The requirements of the sand are not strict. Very fine grains and clay are objectionable. One company employs sand that will all pass 20 mesh and 90 per cent of which is retained on the 150 mesh.

*Banding Sand.*—This sand, which is used for second or semifinal grinding of plate glass, is considerably finer than that employed for the first grinding. It is less used than formerly, as artificial abrasives have replaced it to a large extent.



*Sandblast Sand.*—This type of sand, which by means of an air blast is blown against the surface to be abraded, is used in the foundry for cleaning castings, for removing paint, rust or stain from old surfaces, for carving designs on stone and other purposes. The material used for sandblasting is commonly obtained from sedimentary beds of varied origin, but volcanic sand has sometimes been employed.

TABLE 9.—*Different Grades of Sandblast Sand*<sup>a</sup>

Retained on Mesh	Percentages			
	Sand 1	Sand 2	Sand 3	Sand 4
4				2.70
6			0.00	76.60
8			1.55	99.80
10		0.00	53.15	100.00
14	0.40	32.20	84.25	
20	35.70	78.35	99.70	
28	97.20	92.05	100.00	
35	99.90	98.20		
48	100.00	99.70		
65				
100				

<sup>a</sup> All from Cape May, N. J.<sup>21</sup>

Blast sands may be separated into different grades, from fine No. 1 to coarse No. 4. Table 9 sets forth their differences. The grade numbers are not to be regarded as accepted standards. A general range of size is: No. 1, through 20 mesh, retained on 48 mesh; No. 2, through 10 mesh, retained on 28 mesh; No. 3, through 6 mesh, retained on 14 mesh; No. 4, through 4 mesh, retained on 8 mesh.

There is no standard requirement regarding the shape of grains of blast sands. Some advocate sharp grains because they are supposed to cut faster, but others believe that rounded grains last longer. Cole<sup>7</sup> found that rounded grains used on steel plates did not grind as smooth as angular ones when the nozzle was held at an angle to the plate.

TABLE 10.—*Production of Grinding and Polishing Sand*<sup>a</sup>

Year	Short Tons	Value per Ton (F.o.b.)	Year	Short Tons	Value per Ton (F.o.b.)
1926	1,859,215	\$1.35	1931	607,589	\$1.82
1927	1,686,762	1.30	1932	419,691	1.52
1928	1,538,046	1.30	1933	710,000	1.50
1929	1,636,464	1.41	1934	571,191	1.82
1930	1,115,915	1.45	1935	900,000	1.40

<sup>a</sup> Minerals Yearbook, U. S. Bureau of Mines.

## ENGINE SAND

Engine sand is used to prevent the driving wheels of engines or the wheels of streetcars from slipping. The requirements are a minimum of fine particles. Either round or angular grains are satisfactory. Different railroads have different specifications, but one specified by the Pennsylvania Railroad system calls for clean silica sand between 20 and 80 mesh.<sup>21</sup> A noncaking quality is important to ensure free running of

TABLE 11.—*Production of Engine Sand*<sup>a</sup>

Year	Short Tons	Value per Ton (F.o.b.)	Year	Short Tons	Value per Ton (F.o.b.)
1926	2,355,399	\$0.61	1931	1,604,123	\$0.63
1927	2,618,890	0.63	1932	1,151,011	0.60
1928	2,413,043	0.64	1933	1,150,000	0.62
1929	2,318,931	0.69	1934	1,250,000	0.66
1930	1,733,204	0.69	1935	1,700,000	0.66

<sup>a</sup> Minerals Yearbook, U. S. Bureau of Mines.

the sand. With the decrease in the use of trolley lines, it is probable that the use of traction sand is decreasing.

## SAND FOR SHEET ASPHALT

This sand is used in construction of the surface course of asphalt pavements, and bituminous-concrete pavements in which sand predominates. The specifications (A.S.T.M.) call for grains that are clean, tough, rough surfaced, angular and free from clay. An asphalt mixture shows an average composition of sand, 75 per cent; filler 15 per

TABLE 12.—*Grading of Sand for Sheet Asphalt*

Passing	Retained on	Per Cent	Passing	Retained on	Per Cent
Mesh	Mesh	0-5	Mesh	Mesh	
200	200	6-25	30	40	8-25
100	100	6-25	20	30	5-15
80	80	5-40	10	20	5-15
50	50	5-30	10		95 to 100
40			Inch:		
			1/4		

cent and asphalt, 10 per cent. The amount of filler required will be affected by the size of the sand grains. The requirements for grading are given in Table 12.

FIRE OR FURNACE SAND<sup>21</sup>

Silica or sand is used to line furnace bottoms and walls, particularly in open-hearth steel furnaces. It should be high in silica with a small amount of bonding material. Sand with as little as 80 per cent silica is sometimes used. If the sand lacks bond, plastic fire clay may be added.

A scattering of the different grain sizes is desired, so that the finer ones fill the voids between the coarser. They also promote sintering of the bond. Furnace sands in general are similar to fine steel-molding sand. The localities that supply furnace sands are much the same as those from which steel sands are obtained. Pennsylvania, Ohio, New Jersey and Illinois are therefore large producers.

TABLE 13.—*Production of Furnace Sand*<sup>a</sup>

Year	Short Tons	Value per Ton (F o b )	Year	Short Tons	Value per Ton (F o b )
1926	434,541	\$1 06	1931	88,189	\$1 49
1927	410,801	1 10	1932	36,698	1 48
1928	305,659	1 18	1933	95,000	1 20
1929	440,679	1 10	1934	137,000	1 24
1930	258,241	1 29	1935	160,000	1 38

<sup>a</sup> Minerals Yearbook, U. S. Bureau of Mines.

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## CHAPTER XLII

### DIMENSION STONE

BY OLIVER BOWLES,\* MEMBER A.I.M.E.

**D**IMENSION stone is a convenient term applied to stone sold in blocks or slabs of specified shapes and usually of specified sizes, as contrasted with crushed, broken and pulverized stone, discussed in another chapter. Dimension stone includes cut stone, rough building stone, ashlar, rubble, paving blocks, curbing and flagging. Riprap—large, irregular blocks used for shore protection—is classed more properly with crushed and broken stone.

#### COMPOSITION, VARIETIES AND PROPERTIES

Stones used as blocks or slabs are of many kinds. The principal varieties are granite, sandstone, limestone and marble. Slate is covered in a separate chapter. Granite is an igneous rock consisting essentially of feldspar, quartz and mica. Limestone is calcium carbonate, with which may be associated certain impurities such as clay, sand, or iron oxide. Dolomites and magnesium limestones used as dimension stone are classed with the high-calcium limestones, as they are used in the same way. Marble is a crystalline form of limestone. Sandstone consists essentially of silica in the form of sand grains cemented together into a coherent mass.

Although rock occurrences are numerous and widespread, the exacting requirements of use condemn all but a small fraction of them for commercial use as dimension stone. No deposit that has closely spaced cracks, lines of weakness, or joints can be so used because sound blocks of moderate to large size are demanded. Uniform texture and grain size, constant and attractive color, and freedom from impurities that may cause stains or deterioration usually are required also.

Hardness is a variable property. Granites are as hard as or harder than steel, while limestones and marbles may be scratched easily with a knife. The hardness of a sandstone depends upon the degree of cementation of the grains. Workability depends to a considerable extent on hardness, but the degree of hardness has little influence on use except when stones are subjected to severe abrasion, as on steps or floors.

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"Texture" as applied to stone relates to the arrangement, uniformity, and size of the constituent grains.

Stones are of many colors and thus can satisfy a variety of tastes. For monumental stone a marked contrast between polished and tooled surfaces is preferred in order that inscriptions may be read easily.

Strength requirements usually may be disregarded because any sound structural stone suitable in other respects is almost invariably many times as strong as would be required for any ordinary use. The pressure on the base course of the Washington Monument is less than 700 lb. per sq. in., and high-grade granites, limestones, sandstones and marbles will sustain a crushing load of 10,000 to 25,000 lb. per sq. in. A structure made of stone with a crushing strength of 15,000 lb. per sq. in. would have to be nearly  $2\frac{1}{2}$  miles high before crushing of the lower courses would occur. However, stone disintegrates more readily under severe stress and therefore a safety factor of 20 is demanded usually. A crushing strength of 5000 lb. per sq. in. is considered satisfactory for all ordinary uses.

Porosity, expressed as the percentage of pore space to total rock volume, ranges from 1 to 10 per cent for commercial sandstones and from 0.5 to 5 per cent for limestones, while marbles, granites and slates may run lower than 0.1 per cent. The specific gravity of common rocks ranges from 2.2 to 2.8 and the weight per cubic foot from 140 to 180 lb. Details of the physical and chemical properties of stones from many localities are given by Parks,<sup>25</sup> Merrill<sup>22,23</sup> and Kessler.<sup>16</sup>

#### ORIGIN, OCCURRENCE AND DISTRIBUTION OF DEPOSITS

Igneous rocks are those that originated from molten masses or magmas. Slow cooling of these magmas deep within the earth resulted in coarse crystallization of the constituent minerals. The principal rock type of this class is granite. Magmas that cool more rapidly form finer grained rocks, such as basalts and aplites.

Sedimentary or stratified rocks are those laid down in strata or layers. The materials forming them are derived from the disintegration of pre-existing rocks. Vast quantities of the products of rock decay are carried by water and ice and deposited as sediments in oceans, lakes and river beds. In addition to this detrital material millions of organisms that inhabit oceans or lakes secrete calcium carbonate or silica from the water to form their shells and their skeletal remains add to the accumulation. The principal rocks thus formed are sandstone, limestone and shale, but the latter, because of its easy disintegration, is not used as dimension stone.

Most sedimentary rocks used commercially as building stone have suffered little deformation and therefore occur as horizontal or nearly horizontal beds. In many places, however, the great mountain-building

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<sup>25</sup> References are at the end of the chapter.

forces ever at work within the earth have squeezed, crumpled, and folded the sediments. Heat and pressure may bring about a rearrangement and crystallization of the constituent minerals, forming new types known as metamorphic rocks. Thus, limestones are converted into marbles and sandstones into quartzites. Marbles are used widely for structural and ornamental purposes but quartzites, on account of their excessive hardness, are used sparingly. Slates, covered in another chapter, are formed by intensive metamorphism of shales.

The Appalachian district of eastern United States extending from Maine and Vermont to Georgia is a rugged, mountainous region in which igneous and metamorphic rocks abound. There are important deposits of granite, marble and slate in this territory. The great area lying between the Appalachian belt and the Rocky Mountains is characterized chiefly by nearly horizontal sediments that are well represented by the famous limestone deposits of Indiana and the well-known sandstones of Ohio. Important granite outcrops also occur in this area, notably in Minnesota, Wisconsin, Missouri and Texas.

In the Rocky Mountain belt numerous igneous and metamorphic rocks occur, and from this belt to the Pacific Coast igneous rocks are most abundant. Limestones also occur and have been metamorphosed into marbles in places. Vulcanism of comparatively recent geologic age has produced lavas, rhyolites, and volcanic tuffs that rarely occur in the Eastern or Central States.

Building and ornamental stones abound in many foreign countries. Notable examples are the marbles of Africa, Italy, France, Belgium and England; the granites of Scotland, Finland, Norway and Sweden; the granites and marbles of Canada; the travertine of Italy; and the onyx marble of Mexico. Many of those occurring in the Old World were used in ancient times for building pyramids, obelisks, temples, and other classic masterpieces of Greek, Roman, and Egyptian art and architecture. Although many are now in ruins, they furnish valuable data on the culture and mode of life of ancient peoples.

#### POLITICAL AND COMMERCIAL CONTROL

Stone deposits for the most part are owned and operated by the nationals of the respective countries in which deposits occur. Most countries in an emergency would be self-sufficient, although under normal conditions of trade a good deal of stone crosses international boundaries. Many varieties have no counterparts outside their own restricted areas, and on this account they are conveyed long distances to satisfy the demands of architects, builders, and dealers in memorials.

#### PRODUCTION AND CONSUMPTION

Statistics of the stone industries of the United States are based on sales. Stocks of finished products on hand are an unimportant factor;

therefore production and consumption may be regarded as virtually equal. Table 1 shows sales of dimension stone during certain representative years.

TABLE 1.—*Sales of Principal Varieties of Dimension Stone in United States during Certain Representative Years<sup>a</sup>*

	1929		1932		1934	
	Short Tons	Value	Short Tons	Value	Short Tons	Value
Granite . . .	1,711,030	\$23,369,396	648,420	\$11,743,408	543,360	\$ 8,685,501
Marble . . .	553,660	16,545,312	179,130	7,297,291	81,720	3,194,882
Limestone . .	1,703,240	21,501,201	640,780	7,112,532	585,510	3,621,132
Sandstone . .	655,600	5,286,707	172,180	1,524,222	106,900	978,941
Total . . .	4,623,530	\$68,702,616	1,640,510	\$27,677,453	1,317,490	\$16,480,456

<sup>a</sup> Minerals Yearbook, U. S. Bureau of Mines

Fig. 1, based on statistics of the United States Bureau of Mines, shows trends in sales of building and monumental stone during recent years.

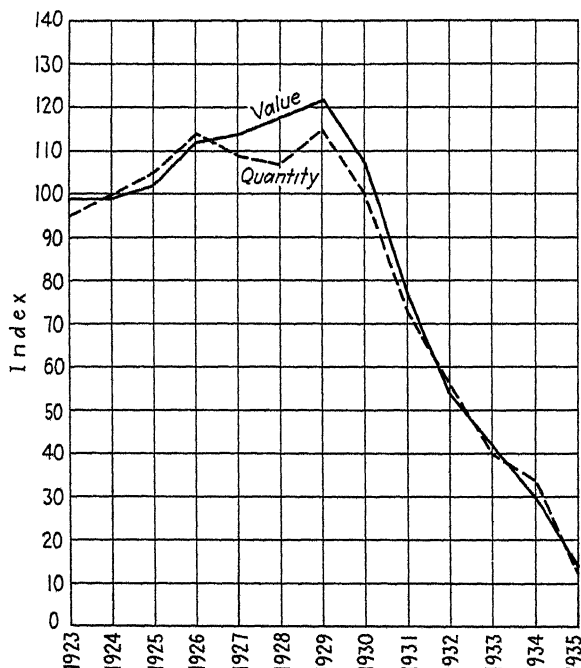


FIG. 1.—BUILDING AND MONUMENTAL STONE SOLD IN THE UNITED STATES, 1923-1935.

Fig. 2 shows trends in sales of building stone, by kinds, for a series of years. The unprecedented low level that such sales have reached since 1931 reflects the stagnation in building construction. Detailed statistics

of the dimension-stone industries are published each year by the United States Bureau of Mines in the Minerals Yearbook.

### PROSPECTING, EXPLORATION AND MINING

A small fraction only of the innumerable rock deposits scattered in every part of the country yields suitable dimension stone; therefore very careful exploratory work should be done to assure a sufficient supply of satisfactory material before money is spent by a prospective operator for development. If the rock appears in bare outcrop the exposed surface should be examined carefully, and enough rock should be removed to permit examination of a fresh surface below the zone of weathering.

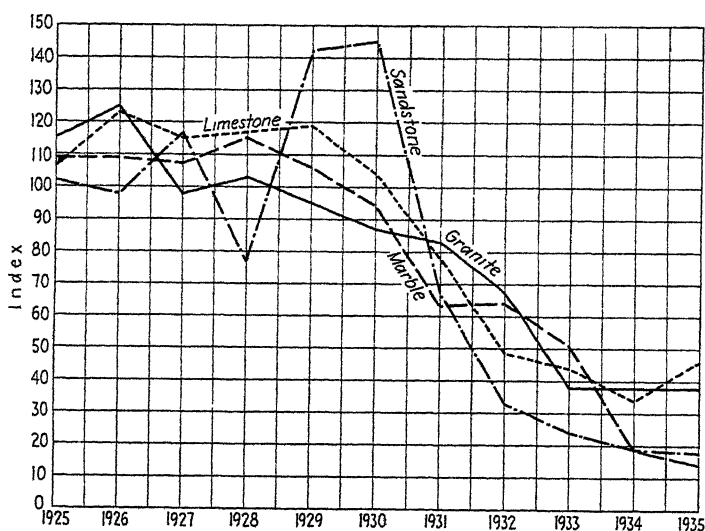


FIG. 2.—QUANTITY OF BUILDING STONE SOLD IN THE UNITED STATES, 1925-1935, BY KINDS.

The features to which most attention are given are freedom from cracks or closely spaced joints, uniformity of texture, attractiveness of color, and for some uses, capability of taking a polish.

Surface exploration should in most instances be supplemented by core drilling. The drill cores are usually 3 in. or smaller in diameter. In flat-lying beds prospect drilling is simple and the holes may be widely spaced—100, 500 or even 1000 ft. apart. Where beds are folded or faulted a thorough study of the structure should be made with the aid of geologic maps. The position, thickness and slope of the beds is first determined and the drill holes usually are sunk at right angles to the beds. Accurate records are made of each hole, and as the core sections are removed they are marked, recorded and stored for future study.

Granite deposits usually are fairly uniform over considerable areas and their depth, with few exceptions, is very great. Generally, therefore,



prospect holes may be widely spaced. Core drilling with shot or diamond drills costs \$3 to \$5 a foot, depending on the nature of the rock and drilling conditions.

*Stripping.*—Stripping is the process of removing the overburden of clay, gravel or sand from the rock surface. If a considerable volume of material is to be moved mechanical methods usually are employed. The hydraulic method is the most economical but it can be used only where an ample supply of water is available, where a waste-disposal area is situated favorably, and where the overburden is friable enough to be washed down and carried away with a stream of water. Dragline scrapers or excavators, power shovels, clamshell buckets operated from derrick arms, tractor excavators and scrapers are other types of mechanical equipment used. For removal of thin overburden, particularly on rugged rock surfaces, hand methods must be used. The cost of power-shovel stripping ranges from 15¢ to 50¢ a cubic yard, depending upon working conditions and nearness of disposal area.

*General Methods of Operation.*—Two general methods of operation are followed in securing dimension stone—open-pit quarrying and underground mining. Open pits are of two types, the “shelf” quarry and the “pit” quarry. Where the ledge of stone forms a hill the floor of a quarry worked in the hillside may be little if any lower than the surrounding country. Such an opening is known as a shelf quarry. Both transportation and drainage are favorable in such quarries. Pit quarries are more common. They are sunk below ground level, access is gained by stairs, ladders or mechanical hoists, and material is conveyed from the quarry by inclined tracks, derricks or cableway hoists. Pits may reach depths of several hundred feet.

If the beds of serviceable stone dip beneath waste rock, or if for any other reason open-pit work is not feasible, underground methods may be pursued. Certain marble operations in Vermont and Alabama have extensive underground workings. Selective mining can be accomplished best by the underground method; no stripping is required and workers are not exposed to the weather. On the other hand, the cost of making primary openings is high, and much material must be left for roof support. A method of quarrying known as “undercutting,” intermediate between the open pit and the tunnel, is sometimes pursued. Channel cuts along the walls have an outward slant, thus the floor space is enlarged gradually. Wings or buttresses of stone may be left at intervals for wall support.

## GRANITE

Blocks of granite are produced in four chief areas: (1) the Appalachian district of the eastern United States from Maine to Georgia; (2) the Middle Western States, particularly Minnesota and Wisconsin.

sin; (3) the Rocky Mountain States; and (4) the Pacific Coast States, particularly California.

The five leading states in order of value of production of building granite in 1934 were Massachusetts, Minnesota, New Hampshire, Maine and Georgia, and of monumental granite, Vermont, Georgia, Wisconsin, Minnesota and South Dakota.

*Vermont.*—Granite is produced in Vermont chiefly in the district surrounding Barre and Graniteville, Washington County. The typical "Barre" granite, which is widely used for monuments, is a fine to medium grained gray to white biotite granite. The darker varieties are most in favor for monument dies and the lighter for buildings, mausoleums and monument bases. Other important centers are Newark, Hardwick and Groton, Caledonia County; Derby, Orleans County; Dummerston, Windham County; and Bethel, Windsor County.

*Massachusetts.*—The most important center for monumental granite is at Quincy, Norfolk County. Quincy granite is medium to coarse grained, and ranges in color from greenish gray to dark bluish gray. Building granites are produced chiefly at Milford, Worcester County; West Chelmsford and other towns in Middlesex County; Becket, Berkshire County; and Fall River, Bristol County.

*New Hampshire.*—Granites for both building and monumental uses are quarried near Redstone and Conway, Carroll County; Milford, Hillsborough County; and near Concord, Merrimack County.

*Maine.*—Paving-block manufacture is the principal branch of the granite industry in Maine. This state produces about 40 per cent of the total output of granite paving blocks in the United States. Except for important quarries at Hallowell, Kennebec County, and North Jay, Franklin County, the industry is centered in the vicinity of Penobscot and Bluehill Bays along the eastern shore.

*Georgia.*—Building granite, paving blocks and curbing are produced at Stone Mountain and Lithonia, De Kalk County, and memorial granite near Elberton, Elbert County.

*Minnesota.*—"St. Cloud red" and "St. Cloud gray" are well-known memorial granites quarried near St. Cloud, Stearns County. A coarser grained granite quarried near Rockville is used chiefly as building stone. Granite is quarried also at points along the Minnesota River Valley.

*Wisconsin.*—Granites of a variety of colors ranging from black, gray, and pink to brilliant red are quarried in Wisconsin. The chief centers are Mellen, Ashland County; Berlin, Green Lake County; Wausau, Marathon County; Amberg, Marinetta County; Montello, Marquette County; and Waupaca, Waupaca County.

*Other States.*—Other active granite centers are Mount Airy and Salisbury, N. C.; Rion, S. C.; Ausable Forks and Peekskill, N. Y.; Westerly, R. I.; Groton, Millstone, and other points in New London County,

Conn.; Roedey, Coopersburg, and St. Peters, Pa., where "black granite" (diabase and gabbro) is produced; Milbank, S. Dak.; Llano, Marble Falls, and Fredericksburg, Tex.; Salida, Colo.; Mountain Park, Granite, and Roosevelt, Okla.; Graniteville, Mo.; Raymond, Porterville, Perris, Lakeside, Nevada City, and Chilcoat, Calif.; and Medicine Lake, Wash.

### STRUCTURAL FEATURES

Although innumerable granite deposits occur throughout the country, only a relatively small number of them are of suitable color or texture or are available in sound blocks large enough for use as dimension stone. Most granite deposits are characterized by joints or seams—natural fractures that cut the rock mass usually in a nearly vertical direction. They tend to occur in more or less parallel arrangement, frequently in two major systems intersecting approximately at right angles. If they are spaced closely the rock is useless for dimension stone, but if regularly spaced 10 to 30 ft. apart they are of great advantage in quarrying. Horizontal partings that separate the granite into sheets or layers are known as sheeting planes.

Most granites split in some directions with greater ease than in others. The direction of easiest splitting is known as the "rift." A second less strongly marked tendency to split, usually at right angles to the rift, is known as the "grain" or the "run." The direction at right angles to both rift and grain is called the "hard way" or "head grain." These splitting directions are obscure and can be recognized only by skilled stonecutters, but they are of the utmost importance in processes of quarrying and shaping blocks of granite.

At some period in its history the granite mass may have been intruded by a rock magma injected into joints or fissures. Such "dikes" are of two kinds—light colored, known as "aplite," and dark colored, consisting of basalt or diabase. Dikes are to be avoided in quarrying and if they are numerous the deposit may be worthless. "Knots," consisting of dark spots, lenses or patches, and "hair lines," fine lines of discoloration that may be either veins or dikes, are also objectionable, particularly on granite to be polished.

### QUARRY METHODS

The position and direction of quarry walls usually are governed by the joint systems, because an open joint usually forms a "heading" or quarry wall. Fortunately the rift usually parallels a major joint system.

Drilling is the most important quarry process. Tripod piston drills widely used in early years have been displaced generally by hammer drills, which can be moved more quickly and handled more easily. For making rows of closely spaced holes the bar drill is used. This consists of a horizontal bar 12 to 14 ft. long supported by a pair of steel legs at each end. A heavy hammer drill is mounted on the bar and may be moved

quickly to any desired position by means of a pinion working in a rack of cogs. At Barre, Vt., each bar-drill machine averages 100 to 120 lin. ft. per day for moderately deep drilling, using a  $2\frac{3}{4}$ -in. bit as a starter. For shallow holes hammer drills held in the hands usually are employed.

In opening a new bench on a quarry floor, the most difficult step is to obtain a free face from which to work. A mass of rock 3 to 5 ft. wide and the depth of the bench must be removed to give the necessary working space. Different methods are employed to make such a trench. If the mass is flanked on either side by an open seam it may be removed by drilling and blasting. If open seams cannot be utilized thus, holes may be drilled in two parallel rows 3 to 5 ft. apart and the intervening rock shattered with dynamite and removed as waste. Another method is to drill the holes close together and remove the thin walls or webs between them with a flat broaching tool, thus forming a narrow channel. Two such channels may be made parallel with each other and any desired distance apart. When the rock between them is removed a trench is provided. This method is less wasteful than blasting, for the mass of rock between the channel cuts can be removed in block form and utilized. In some quarries it is advantageous to use the channeling method for many subsequent cuts. Although this method is more costly than blasting, the waste reduction is more than sufficient to compensate for the additional cost.

The larger masses may be separated from the solid ledge by blasting with light charges of black blasting powder. To facilitate splitting, fractures are made in the directions of rift and grain as far as possible. Subdivision of the large blocks is accomplished by "plug-and-feather" wedging. Holes  $\frac{3}{4}$  in. in diameter and 4 or 5 in. deep are drilled in line 6 to 18 in. apart. Pairs of soft-iron plates with steel wedges between them are placed in the holes and sledged lightly back and forth until a fracture is made. If a break is to parallel the hard way "foot holes" 1 to  $1\frac{1}{2}$  ft. deep are drilled  $1\frac{1}{2}$  to 4 ft. apart, with shallow "plug holes" between them. To obtain large, thin blocks suitable for platforms or mausoleum covers, holes are drilled in line about 6 in. apart to almost the full depth of the block and a fracture is made by driving wedges into them.

Blocks may be removed from wide, shallow quarries with tractors, auto trucks, or two-wheeled mule carts. Derricks usually are employed for the deeper, narrower quarries.

#### PREPARATION FOR MARKET

The principal granite products are monumental stone, building stone, paving blocks, curbing and rubble. The term "rubble" is applied generally to rough, irregular building stones having one good face.

Curbing and paving stones usually are shaped on the quarry floor or in an adjacent yard. Curbstones are of two kinds, straight and corner

(curved). The latter are the more difficult to make and require more stone and more time and labor. After curbstones are split to approximate size and shape they are smoothed to some extent with hand tools and finished with pneumatic tools. The part that appears above the pavement when the curb is placed is dressed to a smooth surface, but the part remaining underground may be much rougher. Specifications for size and surfacing differ in various cities.

In paving-block manufacture, the larger blocks are subdivided by driving plug-and-feather wedges into shallow drill holes, and the directions of rift and grain are followed carefully. Final subdivision often is made with a "bull wedge," a blunt steel plug placed between two iron feathers in a shallow notch cut with a pneumatic tool. One blow with a sledge will make the break. Paving stones are made in various sizes. The smaller ones are known as Belgian blocks. Market quotations in New York usually specify 30 blocks to a square yard.

In early years quarry blocks were dressed entirely with hand tools. These are used still for certain operations, but most of the work is done with mechanical tools. The pneumatic tool, with which much smoothing and carving is now done, is guided by hand but the impact is supplied by compressed air.

The "roughing down" of surfaces to a comparatively uniform smoothness is accomplished chiefly with a surfacing machine that consists of a cutting head mounted on a horizontal swinging arm. Cutting tools fitted into the nose of the cutting head are driven against the stone by rapid blows of an air-driven piston hammer. As the cutter is guided over the surface it chips off fragments and gradually works the stone to a smooth and even surface. By using different kinds of tools different kinds of surface can be obtained. A four-point tool with a square face on which are four blunt projections gives a rather rough surface. A bush-hammered surface is obtained with a tool consisting of a series of parallel steel plates.

Carving requires much skill and experience. Air-driven tools in great variety are used. A large part of the carving formerly done with hand tools is accomplished now by sand blasting. The rock surface is first coated with a molten rubberlike compound known as "dope," which hardens quickly. Letters or other designs are imprinted on the surface, and with a small sharp tool the dope is removed from all parts that are to be cut below the surface. Powdered carborundum or sand is driven against the surface from a nozzle carrying air pressure of 80 to 100 lb. per sq. in. The sandblast cuts away the stone from all unprotected surfaces. Intricate designs may be fashioned in great detail in a mere fraction of the time required for hand carving.

During recent years sawing has become an established process in granite mills. The gang saw consists of a series of steel blades set in

parallel position in a frame that has a forward and backward motion. Spacing of the blades determines the thickness of the slab or block. Blades that are about  $\frac{1}{2}$  in. thick and have notches about one foot apart on the lower edge are fed with steel-shot abrasive. The cutting rate ranges from 4 to 9 in. per hour.

Circular saws 5 to 12 ft. in diameter and provided with detachable notched steel teeth also have been designed for granite cutting. The granite blocks are mounted end to end on cars and the spaces between filled with plaster of Paris to keep the shot in the cut as the saw passes from one block to another. The rate of travel ranges from  $1\frac{1}{4}$  to 5 in. a minute, and the saws cut from 25 to 100 sq. ft. per hour. Sawing is costly, but it conserves stone and, as cuts can be made in any direction irrespective of rift or grain, the most attractive colors and patterns attainable may be secured. Also, the surfaces are so smooth that rubbing and polishing are facilitated greatly.

Much memorial and ornamental building granite is polished. Blocks pass through several successive stages of manufacture before a polished surface is obtained. Sawed blocks, or those smoothed with surfacing machines, are set in groups with their upper surfaces on an even plane. They are boxed in and all cracks filled with plaster of Paris. A power-driven rotary head known as a "scroll," consisting of iron rings or segments, is supplied with steel shot and when guided over the surface wears it smooth. This process is known as "ironing." The next step is to use a lighter scroll with Carborundum powder as abrasive. Finer and finer grades are used in succession. This process is known as "emerying." The final stage, known as "buffing," consists in polishing the surface with revolving pads of paper-mill felt supplied with "putty powder"—fine-grained tin oxide. Single blocks are now polished in some mills without setting in plaster of Paris. The abrasive is collected in a trough for re-use. Small air-driven portable polishers are used for narrow edges, and curved or irregular surfaces are polished by hand.

The principal surface finishes are "rock-faced," "hammered," "steeled" (obtained by "ironing" with steel shot), and "polished."

Silicon carbide wheels are now used generally in granite-cutting establishments to cut moldings, to shape fluted columns, and for similar processes. For making columns the granite block is first dressed to cylindrical shape and then placed in a lathe. Steel disks chip off projections and gradually work the stone down to a smooth surface. It is then ground with steel shot and Carborundum powder, and polished in the usual way.

## LIMESTONE

Limestones are of many kinds. Some are named after their principal impurities, for instance "siliceous," "ferruginous," and "carbonaceous."

"Oolitic" is a name applied to those consisting of small rounded grains resembling fish roe. Limestones also are named after the characteristic shells of which they are composed, such as "coral," "crinoid," and "coquina." Travertine, a product of chemical precipitation from hot springs, is classed with limestones.

Limestones occur in every state, but only a relatively small number of the deposits are suitable for use as dimension stone, or are favorably situated for quarrying or marketing. The well-known Indiana deposits furnish a large share of all sales.

*Alabama.*—Oolitic limestone is quarried extensively near Rockwood, Franklin County. The quarries and mills are well equipped and the stone has a wide market.

*Florida.*—Coquina and other shell limestones are abundant in Florida, and are quarried for building purposes near Volusia, Volusia County; on Windly's Island, Monroe County; and at New Port Richey, Pinellas County.

*Indiana.*—Indiana oolitic limestone is one of the most widely known building stones. This state produces 80 to 85 per cent of all the building limestone sold in the United States. More than 20 companies operate 30 to 40 quarries and mills, and about a dozen more operate only finishing mills. The limestone used occurs in a massive bed 25 to nearly 100 ft. thick, which extends north and south for a distance of about 125 miles. It is tilted gently westward with a dip of 34 to 70 ft. per mile. It is of two principal colors, buff and gray. When first quarried it is comparatively soft and easy to work but it becomes harder upon exposure to the weather. Its workability is one of its principal assets. It is an attractive, durable stone that is used widely both for public buildings and in private construction. The chief centers of production are in the Bedford-Bloomington district in Lawrence and Monroe Counties. Some stone is quarried at St. Paul, Decatur County, and at Romona, Owen County.

*Kentucky.*—An oolitic limestone similar to that produced in Indiana is quarried near Bowling Green, Warren County. It occurs in sound beds 10 to 20 ft. thick.

*Minnesota.*—Yellow and pink limestones quarried near Kasota, Le Sueur County, and Mankato, Blue Earth County, are popular for interior decorative building. Some of the stone will take a polish and is classed as marble. Blue and buff limestones quarried near Mankato are well adapted for heavy masonry. Architectural limestones are quarried near Mantorville, Dodge County, and Winona, Winona County, also.

*Texas.*—Texas limestones have attained some prominence during recent years. The principal quarries are at Cedar Park, Williamson County; Lueders, Jones County; and Del Rio, Kinney County.

*Colorado.*—Travertine that compares favorably with the Italian stone is quarried near Salida, Chaffee County. It is used for both interior and exterior building. A green siliceous limestone is quarried near Manitou, El Paso County.

*Other States.*—Building limestones are quarried also near Quincy, Alton and Joliet, Ill.; at Silverdale and Manhattan, Kans.; and at Wauwatosa and Lannon, Wis. Travertine is produced near Gardiner, Mont.

### QUARRY METHODS

Most limestone deposits used for dimension stone are nearly flat lying and of limited thickness. Almost invariably the quarries are of the open-pit type. As the stone is worked out, the waste is thrown into the abandoned pits. Where usable beds are thin much stripping may be required because the quarry must be widened constantly. If inferior stone lies over the good beds, it also must be removed, and sometimes the cost of removal is high. At some of the Indiana quarries the overlying waste rocks form a continuous bed with the good stone. Blasting the waste rock, therefore, is impractical as it would damage the good stone beneath. On this account the waste rock must be removed by channeling and wedging, which is more costly. At other quarries an open seam separates the defective from the usable stone, and the waste rock can be removed safely by blasting.

The first step in the process of block removal is channeling. The channeling machine is mounted on a frame and travels back and forth on a track. Steel bars operating with a chopping action similar to that of a reciprocating drill cut a narrow channel in the rock. Some are driven by steam and some by electricity. Channel cuts are made about 4 ft. apart and 8 to 12 ft. deep. The duplex machine, which makes a double cut, averages 200 to 300 sq. ft. of channel a day and the cost of cutting is 8¢ to 12¢ per square foot.

The high cost of channeling has encouraged operators to develop more economical methods. Wire saws have been tried successfully in several quarries. The wire saw consists of a three-strand steel cable of about 1/4-in. diameter. It runs as a belt and where it comes in contact with the rock it is fed with sand and water. It cuts rapidly, requires little power or attention, and reduces the waste greatly. It offers so many advantages that one would expect to find it in much wider use than it is at present.

When a floor is channeled a key block is broken loose by driving wedges in the channel cut, and when it is removed space is provided for drilling beneath adjacent blocks. Wedges are driven in the drill holes to break the blocks free at the floor. When a wide floor space is obtained successive masses of stone, each about 50 or 60 ft. long, 4 ft. wide and 12 ft. high, are turned down on the quarry floor, where they are sub-



divided into mill blocks by driving wedges into small shallow drill holes. Common mill block sizes are 10 by 4 by 3 ft. and 10 by 4 by 4 ft. The blocks are hoisted from the quarry with steel or wooden derricks and are either piled in storage within reach of the derrick arm or are loaded on railroad cars and taken to a storage yard or to a mill for fabrication.

#### PREPARATION FOR MARKET

Much limestone is sold as mill blocks and such blocks must have smooth faces and must be trimmed to true rectangular shape. The process of trimming blocks is known as "scabbling." Various methods of scabbling are employed; it may be done by hand with a scabbling pick, with circular saws, wire saws, heavy iron disks provided with cutting tools, diamond-toothed drag saws, or scabbling planers. The planers consist of massive blades, which scrape the surface. Such blades will remove  $\frac{1}{4}$  to  $\frac{1}{2}$  in. of stone at each cut. Blocks for export trade or for shipment to distant mills are scabbled most carefully.

Where blocks are fabricated into finished products in near-by mills the scabbling step may be omitted. Well furnished finishing mills are equipped for drafting and patternmaking, sawing, planing, jointing, milling, turning, fluting, cutting, carving, packing and shipping. Most limestone fabricated in mills in the quarry districts is furnished on contract for specific jobs and therefore each block or slab is cut for its particular place. Consequently, the preparation of detailed shop drawings and patterns is an important preliminary step. A drawing of the block showing exact dimensions is consulted at each step in the manufacturing process.

Overhead traveling cranes with lifting capacity up to 50 tons handle blocks of stone expeditiously. Heavy powerful cranes are used for large blocks and lighter more rapidly moving cranes for smaller pieces.

The first step is to saw rough blocks into slabs. Gang saws like those described in the preceding section on granite are used generally. They operate with a swinging motion, the blades lifting toward the end of the stroke and permitting the abrasive to wash under them. The abrasives used are sand, "chats" (cherty rock obtained at the Missouri lead and zinc mines), and steel shot. Limestone saws much faster than granite; the blades sink at an average rate of about 6 in. an hour. A straight blade with diamond teeth is used for making single cuts.

Circular diamond saws are used generally for making subsequent cuts. Common sizes are 60-in., having 84 diamond teeth, and 72-in., having 110 teeth. Each saw is designed for a standard speed and should be run at no other. With care a saw may be used for six months to a year without conditioning. The cutting edge must be cooled constantly with a stream of water. The saw travels at a rate of 3 to 16 in. a minute, depending on the depth of the cut. Silicon carbide saws are used also.

A planer is a machine provided with a cutting tool having lateral and vertical adjustment. As the block of stone is carried beneath it on a traveling bed or platen a thin layer of stone is scraped from the surface. Both sides and tops of blocks may be planed to desired dimensions. By using tools of any prescribed pattern, moldings may be cut. Some planers are adjusted to cut curved forms. A Carborundum planer consists of two silicon carbide saws with a drum of smaller diameter between them. They trim both top and sides of the block with one motion.

Lathes similar to those used in machine shops are used for turning columns or balusters. Fluted columns are made by first turning the column to a true cylinder, then mounting a fluting tool on the tool post of the lathe. The tool travels back and forth while the column is held stationary. Carborundum wheels are used for fluting also.

A "milling machine" has a rotating cutting head with right-and-left and vertical worm-gear motions. With tools of various shapes and sizes recesses may be cut and blocks of intricate design roughed out so nearly the desired shape that the hand carver's work is reduced substantially.

Carving requires more skill than any other limestone-cutting operation. Most of it is now done with pneumatic tools of many shapes and sizes. Patterns are used for the simpler shapes and plaster of Paris models for the more complicated figures. Many beautiful churches, libraries and other public buildings attest the adaptability of architectural limestone to carving.

Building limestone is sold with various types of surface finish. A tooled surface has fine parallel grooves. A four-cut surface is made with a planer tool having four corrugations to the inch. A bush-hammered surface is rough and pitted. A shot-sawed or ripple surface is deeply scored by using steel-shot abrasive with gang saws. Chat-sawed is somewhat smoother than shot-sawed.

Railway cars are brought into the mill so that the mill hoisting equipment may be used for loading. All blocks are numbered and lettered to show their position in the structure and are packed carefully in excelsior and limestone dust in open-top cars.

### LIMESTONE PRODUCTS

Some companies quarry only and sell the quarry product as rough blocks, or they may have sawmills and sell part of their output in the form of slabs. A second group of companies quarries limestone and manufactures it into finished products. Other companies have no quarries; they buy rough blocks or slabs and manufacture them into finished products. Although cut stone is the chief finished product, increasing quantities of a rougher type of building stone known as "sawed or broken ashlar" are now marketed. It is made in strips 3 or 4 in. thick and in

units of different heights that will combine to give even-range levels. It is well adapted for stone veneer on residences.

### COSTS

Quarry costs range from 20¢ to 30¢ a cubic foot of block stone. The chief item is channeling, which ranges from 8¢ to 12¢ a cubic foot of recovered stone. Milling costs vary greatly, depending upon the amount of work done on each block of stone. Gang-sawing costs 35¢ to 45¢ a cubic foot of finished product. Subsequent sawing costs 12¢ to 15¢ more. Carving adds greatly to the cost. Paper work, including drafting, shop drawings, and patterns, may cost 15¢ to 20¢ a cubic foot on average jobs. The total cost of preparing building limestone with a moderate amount of carving ranges from \$1.50 to \$2.50 a cubic foot.

### SANDSTONE

With few exceptions sandstones consist of quartz grains compacted and cemented together. The four most common cementing materials are iron oxides, clay, calcite and quartz. The last is the most desirable, but if very firmly cemented with quartz the rock becomes a quartzite, which is used little because it is too hard to be worked. Sandstones are sometimes named after their most abundant impurity; for instance, "ferruginous," "micaceous," "calcareous," and "argillaceous." They are sometimes named after their color, as "brownstone." "Bluestone," however, is a term applied to certain thin-bedded or easily cleavable sandstones irrespective of color. Bluestones are also termed "flagstone" and "cleftstone." "Freestone" is a name applied to architectural sandstones that are cut or carved readily with no tendency to split.

The most important use of sandstone is for building purposes, but it has a variety of other uses. Large quantities of curbing and many paving stones are made of sandstone. Flagging is produced for steps, sidewalks and platforms. Grindstones, pulpstones and other abrasive products are made in considerable quantities. Miscellaneous uses include slabs for grave vaults, laundry tubs, electrical switchboards, and lining for acid tanks. Sandstone blocks are used in the same way as silica brick for lining metallurgical furnaces. Quartzites are used as grinding pebbles and for lining tube and ball mills.

*Arkansas.*—A variety of sandstone known as novaculite is quarried at Hot Springs, Garland County, for the manufacture of oilstones, whetstones, and other abrasives.

*Connecticut.*—The well-known "Portland brownstone" has been quarried extensively near Portland, Middlesex County, and has been used extensively in New York, Brooklyn, and other eastern cities. The

spalling observed on many buildings is due to faulty construction—to placing the stone in the wall with its bedding planes vertical rather than horizontal. The stone is durable if properly used.

*Indiana.*—Building sandstone is quarried near Mitchell, Orange County, and at St. Meinrad, Spencer County. Sandstone for abrasive uses is quarried at Floyds Knobs, Floyd County.

*Kentucky.*—Architectural sandstone is quarried at Bluestone and Farmers, Rowan County, and has been quarried at Wildie, Rockcastle County.

*Minnesota.*—Paving stones, building stone, flagging and furnace stone are produced at Sandstone, Pine County. Quartzite produced at Jasper, Rock County, is used for grinding pebbles and to line tube mills.

*New York.*—Paving stones and curbing are the chief products of quarries that have been worked extensively at Albion, Medina, and other points in Orleans County. Bluestone of Devonian age occurs in many places along the Hudson and Delaware Rivers. There are no large centers of production; it is obtained from numerous small quarries.

*Ohio.*—Ohio produces from 50 to 60 per cent of the total output of sandstone in the United States. Blue, gray, buff, and variegated sandstones of Carboniferous age cover a wide area extending from Portsmouth northward to Norwalk and from there eastward to the northeastern corner of the state. The largest quarries that produce stone for building, curbing, flagging, rubble and grindstones are near Amherst, Lorain County, and Berea, Cuyahoga County. Many grindstones and pulpstones have been produced near Empire, Jefferson County, and at Constitution and Marietta, Washington County. A fine-grained sandstone quarried at McDermott, Scioto County, is used as building stone and for burial vaults, grindstones, and small abrasive stones. Variegated architectural stone is obtained near Killbuck, Holmes County. Quarries are operated also in Carroll, Mahoning, and Fairfield Counties.

*Pennsylvania.*—Small sandstone quarries are operated at many points in Pennsylvania, but there are no large quarry centers. Bluestones similar to those in New York are quarried at Pond Eddy and Kimble, Pike County, Meshoppen in Wyoming County, and at other places. A quartz-sericite schist related to sandstone is quarried at Edge Hill, Montgomery County, and sold as a refractory lining for furnaces.

*West Virginia.*—Grindstones and pulpstones are produced near Ravenswood, Jackson County, and at several places in Monongalia County.

*Other States.*—Other sandstone quarry centers are Graystone and Carmel, Calif.; Turkey Creek, Colo.; near Boise, Idaho; East Longmeadow, Mass.; Grind Stone City, Mich.; Raven Rock and Wilburtha, N. J.; Hot Springs, and Sioux Falls, S. Dak.; Crab Orchard, Tenn.; Wilkeson, and Tenino, Wash.; and in Dunn County, Wis.

## QUARRY METHODS

Quarry methods depend on rock structures and on the hardness of the stone. Moderately spaced joints in parallel systems are of great advantage. Quarry walls are maintained parallel with them. Most sandstones used for dimension stone are in nearly flat-lying beds. If open-bed seams are a few inches to 3 ft. apart, the deposit is described as thin-bedded; if they are farther apart, it is thick-bedded; and if the rock is massive, showing no open-bed seams, it is said to be tight-bedded. The rift or direction of easiest splitting is almost always parallel with the bedding. A second less pronounced direction of splitting, known as the run, is usually perpendicular to the rift. It is of great advantage in shaping paving blocks.

The greatest production of sandstone is of the softer types, which may be cut readily with channeling machines. As a rule sandstones so hard that they make good paving blocks are too hard to channel, and primary breaks must be made by blasting.

The channeling process is much the same in sandstone as in limestone, except that the quartz grains wear the steel more rapidly. Steel must be changed frequently. Cuts are about 4 in. wide at the top but become much narrower because of loss of gauge through wear. A stream of water carries away the sand cuttings. Channel cuts are made parallel or at right angles to the major jointing systems. The large masses of sandstone separated by joints or channel cuts may be free at the quarry floor if bed seams are present. If not, a separation is made by driving wedges in drill holes. Subdivision of the larger masses is done by wedging supplemented at times by light charges of black blasting powder.

Bar and hammer drills like those described in the section on granite are used generally. The rate of drilling varies greatly, depending upon the hardness of the stone. In some Ohio quarries, where large grindstones and pulpstones are made, a circle-cutting drill is used. The drill is mounted on a rotating arm and cuts a circular channel.

Small charges of black blasting powder are used for blasting because heavy charges or high explosives would shatter and destroy the stone. The Knox system of blasting is often used. The drill hole is grooved with a flanged tool in line with the direction in which the split is to be made. A plug of cotton waste is placed some distance above the charge and the hole above the plug is filled with sand or other stemming. The air space permits the force of the charge to be exerted over a relatively wide area, which reduces the danger of local shattering. Sometimes air spaces are left both above and below the charge.

The usual plug-and-feather wedges are used for subdividing blocks. For bed-lifting the wedges are usually driven into notches rather than into drill holes. In both blasting and wedging the direction of the break should, if possible, follow the "run" of the rock.

Blocks usually are hoisted from quarries by derricks. The mass of rock worked out from one position of a derrick is called a "motion." The average area of a motion in Ohio is 134 by 61 ft. Grab hooks, chains, and cable slings are used for block attachment.

#### PREPARATION FOR MARKET

*Manufacture of Paving Stones.*—Sandstones in which the grains are firmly cemented together with silica may be used for making paving stones. Paving blocks are shaped either on the quarry floor or in a nearby yard. The process is similar to that described in the section on granite. Directions of rift and run are followed carefully.

*Mill Arrangement.*—Finishing mills for producing building stone, grindstones, curbing and other fabricated products are associated with many quarries. Modern mills are arranged and equipped to promote direct and speedy movement of material through the successive steps of manufacture. A distinctive feature of the better mills is the use of "transfer" and "gang" cars. A block for sawing is placed on a "gang" car. "Transfer" cars are run on a depressed track and have short sections of track across the top. A gang car with a load of finished slabs may be moved quickly from beneath the saws onto a transfer car, taken away, and another transfer car bearing a gang car with a new block may be conveyed immediately to a position in front of the gang saw. Thus a new block can be placed in position for sawing with very little delay.

*Sawing.*—Gang saws similar to those used in granite and limestone mills are generally employed. Gangs with 10 to 15 blades saw average sandstone blocks 5 to 7 ft. long at a rate of 3 to 8 in. an hour when sand is used as abrasive, and 6 to 12 in. an hour when steel shot is used. Subsequent sawing of slabs may be done with Carborundum circular saws or with blades mounted with diamond teeth and set in straight-cut gang frames. Diamond circular saws have not proved satisfactory for sawing sandstone. Wire saws are used in some places for jointing sandstone blocks.

*Rubbing.*—A sand-rubbed surface finish usually is demanded. A rubbing bed consists of a heavy iron disk 10 or 12 ft. across, which rotates in a horizontal plane. The block or slab of stone is held on the surface and when sand and water are supplied the block is ground smooth and reduced to proper dimensions.

*Planing.*—Moldings are cut with planers, usually of the reversible-head type that cut in both directions. For hard sandstones a stream of water must be directed onto the cutting edge to prevent burning of the tool.

*Manufacture of Curbing.*—Curbing is an important sandstone product. The larger blocks usually are split to slabs of the approximate dimensions desired, although massive rock with no pronounced rift may be sawed to

size. Curbing mills are equipped with planers and overhead cranes or pneumatic hoists. Rough blocks are brought into the mill on flat cars, are planed, then reloaded on the same car.

*Manufacture of Grindstones and Pulpstones.*—The stones are cut in circular form in the quarry by means of the circle-cutting drill described previously, or rectangular blocks may be scabbled to circular form. They are then mounted on axles and trimmed to final form with steel bars held against the surface while the stone is rotated.

*Cutting and Carving.*—Building stone is shaped with pneumatic tools and columns are turned in the same manner as in limestone finishing mills.

*The Bluestone Industry.*—The characteristic "cleftstone" occurring along the Delaware, Hudson, and Susquehanna Rivers is usually worked in relatively small quarries and hauled to central shipping points known locally as "docks" situated on navigable water or railway lines. Some of the stone splits with remarkable ease into slabs that may be 10 or 12 ft. square and only 2 in. thick. It was widely used for sidewalks and steps before concrete came into general use. "Edge stone," which splits in thicker beds, is used for curbing, sills, caps, and coping. What is known as "rock" is taken from the more massive beds and is used for building stone.

*Waste Utilization.*—Waste sandstone in large irregular blocks is used as riprap for shore protection, harbor work, or for spillways at dams. Waste also may also be crushed for road stone and concrete aggregate. Sand is an important by-product that is used for sand-lime brick manufacture, for mortar and plaster, for furnace use, or as engine sand.

## MARBLE

Marbles may be classed in three groups. The largest group comprises those derived from limestone by recrystallization resulting from the heat and pressure of mountain-building forces. Exceptionally the recrystallization seems to have been brought about chiefly by circulating water, for there may be no evidence of extreme pressure or deformation of the beds. Marbles may be either high-calcium or dolomitic. The second group comprises the onyx marbles, also called Mexican onyx and cave onyx. They are chemical precipitates of calcium carbonate from cold-water calcareous springs. Usually they show highly ornamental banding. The third group consists of the verd antique or serpentine marbles. They may contain little calcium carbonate but they take a good polish and are used in the same way as are the true marbles. Green is their prevailing color. They may be streaked with white or red and may show ornate patterns.

The principal use of marble is as building stone for both interiors and exteriors. For exterior use marbles must be enduring and free

of impurities that may stain or corrode the surface. For interior use, including floor tile, stair treads, balusters, baseboards and columns, decorative effects are most important. Marble is used also for memorials, statuary and novelties. Onyx marble is particularly adapted for the latter.

As marble results primarily from regional metamorphism, the most important deposits occur in the Appalachian Mountains of the Eastern States and in the Rocky Mountain and coast ranges of the West. Marbles in the Central States, notably in Minnesota, Missouri and Arkansas, have been recrystallized by processes involving little or no deformation of the beds.

*Alabama.*—A bed of marble at least 200 ft. thick occurs near Sylacauga, Talladega County. It is fine grained and mostly white, some beds are of statuary grade. In other beds, layers of light green talc and other silicate minerals give ornamental patterns. The rock is exceptionally pure, running 98 to more than 99 per cent calcium carbonate, and is of very low porosity. It is used for exterior and interior building and decoration and for monuments.

*Alaska.*—White, light blue, mottled, and gray marbles are quarried at Tokeen on Marble Island and at Calder on Prince of Wales Island. The deposits are large and are close to navigable water. Quarry blocks are shipped to finishing mills at Takoma and other points on the Pacific Coast.

*Arizona.*—Onyx marbles occur at Mayer, Yavapai County, and in several other places.

*Arkansas.*—Both gray and black marbles are quarried near Batesville. Independence County, and a coarsely crystallized, light gray marble near Guion, Izard County.

*California.*—A fine-grained dolomitic marble occurring in a great variety of colors is quarried near Lone Pine, Inyo County. Pink, yellow, and gray marbles occur at Columbia, Tuolumne County, and a small output is recorded from Cadiz, San Bernardino County. A decorative marble characterized by red spots and white streaks is quarried at Santa Ana, Orange County. Onyx marbles occur at Suisun, Solano County, and at other points.

*Colorado.*—Pure white and golden-veined marbles are quarried high in the mountains near Marble in northern Gunnison County.

*Georgia.*—Georgia is an important producer of building and monumental marble. The industry is confined almost entirely to the vicinity of Tate and Marble Hill, Pickens County. Highly crystalline white, gray, bluish, and pink marbles are produced in several wide and deep quarries and are fabricated in large, well equipped mills. Most of the Georgia marbles are coarser grained than either the Alabama or the Vermont marbles. Verd antique is quarried near Hollysprings, Cherokee County.



*Massachusetts.*—Dolomitic marbles occur near Ashley Falls, West Stockbridge, and Lee, Berkshire County, but production has been confined to the last locality during recent years. Verd antique is quarried near Westfield, Hampden County.

*Maryland.*—A fine-grained white dolomitic marble has been quarried at Cockeysville, Baltimore County, and fabricated in a mill close to the quarry. Verd antique is quarried at Cardiff, Harford County.

*Missouri.*—Light gray, coarsely crystallized marbles are quarried at Carthage, Jasper County; Phenix, Greene County; South Greenfield, Dade County; and Joplin, Newton County. The beds are virtually undisturbed and show little evidence of regional metamorphism. A decorative fossiliferous marble is quarried at Ozora, Ste. Genevieve County.

*Montana.*—Black marble occurs near Townsend, Broadwater County, and onyx marble near Manhattan, Gallatin County.

*New York.*—Fossiliferous white, gray, pink, and black marbles are obtained near Plattsburg and Bluff Point, Clinton County. Mottled gray and white and solid blue-gray marbles are quarried for both building and monumental use at Gouverneur, St. Lawrence County. A white dolomitic marble used extensively in New York is obtained near Wingdale, Dutchess County. Black marble occurs at Glens Falls and verd antique at Port Henry.

*Tennessee.*—The Tennessee marbles occur in the Holston member of the Chickamauga formation of Paleozoic age. They lie in the valley of the Tennessee River in the vicinity of Knoxville. Seven parallel belts of commercial marble are recognized; namely, Luttrell, Black Oak, Concord, Knoxville, French Broad, Meadow, and Bays Mountain. Many quarries produce light pink and gray marbles used principally for floors, steps, wainscoting, sanitary enclosures, and other interior structural and decorative uses. The stone is used less extensively for exteriors. Tennessee marble is characterized by irregular or zigzag gray, black, greenish, or reddish markings known technically as stylolites but more generally as "crowfoot." Unlike most marbles, those of Tennessee are fossiliferous. Large, well equipped finishing mills are operated in conjunction with the quarries, and much material is shipped also in rough block form to finishing mills in other states.

*Vermont.*—A great marble belt about 80 miles long lies in western Vermont between the Green Mountains and the parallel Taconic Range to the west. The beds are highly folded and produce complex structures that make quarrying difficult. The workable beds have a total thickness of several hundred feet. Marbles in many colors, including white, cream, gray, variegated, green, and blue, are used for exterior and interior building and decoration and for monuments. Large quarries are operated near Dorset, West Rutland, Proctor, Pittsford and Brandon. There are extensive underground operations at West Rutland. Mills

for sawing marble into slabs are situated at West Rutland, Center Rutland, and Florence, and a large marble-finishing mill is operated at Proctor. Reddish siliceous marbles are quarried at Swanton, Franklin County, a black marble on Isle La Motte, Grand Isle County, and verd antique at Roxbury, Washington County, and Rochester, Windsor County.

*Other States.*—White and bluish marbles are quarried and milled at Marble, N. C. Marble is produced at times at King of Prussia, Pa.; Thistle, Utah; and Harrisonburg, Va.

#### QUARRY METHODS

Marble beds are usually tilted and folded, and as they vary in thickness and position from point to point thorough prospecting is required and development must be planned carefully. Both open-pit quarries and underground mines are worked. The most difficult step in underground work is driving the preliminary tunnel or heading. Blasting methods may be used but channel cuts at the floor are essential to prevent the blasts from shattering the marble beds. If the heading is in good marble, ceiling and floor cuts may be made with channeling machines and vertical cuts with a drill mounted on a swivel, a machine known as a Radiolax. Although such cutting processes are slow, marble thus removed is preserved for commercial use. After the heading has been advanced underground operations are similar to those in open quarries except that pillars must be left at intervals to support the roof.

Primary cuts generally are made with channeling machines, which have been described in preceding sections. A favorite machine is the double-swivel channeler, which may be used for both vertical and inclined cuts. Electric air machines are in common use. The rate of channeling varies greatly; recorded averages range from 25 to 80 sq. ft. a day for one machine. The quarry floor may be enlarged by inclining channel cuts outward. This process is known as undercutting.

Wire saws have been tried at several places and some degree of success has been attained in Arkansas and Vermont. The wire cuts slowly and wears out more rapidly than in slate or limestone.

The larger masses are separated from the quarry floor and are subdivided by drilling and wedging. The rift (direction of easiest splitting) usually follows the bedding, but commonly it is not well defined and to secure straight breaks holes must be drilled almost the full depth of the blocks and only a few inches apart. Long wedges are used and some of them are designed to exert pressure throughout the full length of the drill hole rather than at a single point.

Hoisting usually is done with powerful derricks that may have lifting capacities of 15 to 18 tons. The hoist cable may be attached to the marble block by grab hooks, chains, or cable slings.

If marble blocks are to be shipped some distance they are usually "scabbled"; that is, trimmed to true rectangular shape. It may be done by hand with a scabbling pick, by slabbing off the irregular masses with wedges in drill holes, or by cutting with a wire saw.

#### PREPARATION FOR MARKET

The term mill usually is applied to the part of the finishing plant where blocks are sawed into slabs; all other manufacturing processes are classed as shop work. The mill and shop usually are so close together that both may be served by the same overhead traveling crane, although small and large cranes may be operated on the same tracks.

Modern marble mills are equipped with transfer and gang cars as described in the section on sandstone. Gang saws like those used in limestone mills already described are in general use. Marble saws slowly. In stone of moderate hardness the blades may sink at a rate of 1 to 2 in. an hour; in the harder, siliceous marbles they may sink not more than 3 or 4 in. during an entire shift. Some large marble mills are equipped with more than 40 gang saws.

When slabs of marble enter the shop the first processes of manufacture are known as "coping" and "jointing." The former is the process of cutting one slab into two without regard to the finish of the edges. Jointing is the process of cutting true to specified sizes and shapes and with smooth unchipped edges. Carborundum machines generally are employed for this work.

Slabs or blocks may be squared and finished on rubbing beds like those described in the section on sandstone. Irregular surfaces are rubbed by hand with abrasive bricks or with pieces of marble supplied with sand and water.

"Gritting," a process that gives a smoother surface than rubbing, is accomplished usually with abrasive bricks attached to revolving buffer heads. The bricks may be of silicon carbide or aluminum oxide. Gritting produces what is known as a "hone" finish. "Buffing," the process that gives a fine polish to the marble surface, is done with wet "putty powder" (tin oxide) carried on a revolving felt buffer head.

Recutting of blocks and slabs in the shop may be done with circular diamond saws, perforated steel saws using steel shot as abrasive, or with a straight blade in a gang frame. Diamond saws are used most widely. Moldings and cornices are cut with planers, which have been described already.

Carborundum machines are used widely for cutting moldings, balusters, and fluted columns. Columns may be cut with rotating drums fed with steel shot, but generally they are roughed out by hand and then turned in a lathe. The column is first turned to size with a cutting tool,

as in ordinary machine lathe work, ground to a smooth surface with abrasive powders, and polished with putty powder.

Cutting and carving are done principally with pneumatic tools supplemented by sandblast processes, as described in the section on granite.

*Waste Utilization.*—Waste marble blocks may be used as riprap or for rough construction stone, or they may be crushed for road stone and concrete aggregate. Usually marble is low in impurities and so may be used for manufacturing lime or for chemical applications. It may be ground for agricultural use or as filler, or may be pulverized to extreme fineness for "marble flour," which is used as a substitute for whiting.

## OTHER VARIETIES OF STONE

### SOAPSTONE

Soapstone, as the term was used originally, apparently was synonymous with steatite or massive talc. The term as now used relates to all dark gray to greenish talcose massive rocks. Soapstones are metamorphic rocks, generally containing 10 to 80 per cent talc, and the remainder chlorite, amphibole, pyroxen $\acute{e}$ , mica, calcite, dolomite and other minerals.

Soapstone was first used in the United States by the American Indians, who shaped it into bowls, pots, cooking stones, and other objects now seen in many museums. Although a small production has been noted at various times in Maryland, North Carolina, Rhode Island, Vermont, California and Washington, Virginia has always dominated the industry. An extensive deposit in Albemarle, Nelson, and Orange Counties was first developed about 1880. The largest operations are conducted near Schuyler.

The Virginia deposits are regarded as alteration products of basic igneous rocks high in magnesium. They occur as irregular or lenslike dikes 100 to 120 ft. wide, bordered with mica schist and peridotite.

The quarries are 100 ft. long, 100 to 120 ft. wide, and are worked to a depth of about 200 ft. No explosives are used. Cuts are made with channeling machines and, after removal of a row of key blocks, all other channeled masses are undercut to their full depth. The long masses thus set free are subdivided by drilling and wedging in the direction of the natural grain. As the grain dips at angles of 30° to 60° the blocks are roughly diamond-shaped. Average blocks are 4 by 4 by 6 feet.

Milling processes are similar to those in marble mills. Slabs cut with gang saws are trimmed with steel-toothed hand saws similar to those used in wood working. Slab surfaces are smoothed on rubbing beds and are tongued and grooved with Carborundum machines before assembling as laundry tubs, sinks, acid tanks, aquariums, and similar products. An important branch of the industry is the manufacture of

furnace blocks to line retorts in paper mills. Soapstone is used extensively in building construction for wainscoting, sills, caps and spandrels. The harder varieties are used for floor tile and stair treads.

On account of its resistance to chemicals soapstone is used in the manufacture of laboratory hoods, sinks, and tables, photographic laboratory tanks, and similar equipment. It is used for switchboards and other electrical insulation units also. Because of its ability to resist and retain heat it is employed for griddles, footwarmers, and fireless-cooker stones; but these uses are declining.

Furnace blocks and other soapstone products are now being made in Skagit County, Washington, where Carborundum sawing equipment has been designed to make primary cuts in the quarry. Soapstone is quarried for furnace-block manufacture, also, near Thetford Mines, Quebec, Canada.

#### GREENSTONE

A metamorphic rock related in origin to soapstone but consisting essentially of actinolite and chlorite is quarried at Lynchburg, Va. On account of its characteristic color it is called "greenstone." It has an attractive, unfading color, which makes it popular for ornamental building purposes. It has nonskid qualities that recommend it for floor tile and steps. It is used also for making furnace blocks.

#### MISCELLANEOUS STONE

Basalt, gabbro, and other basic igneous rocks are used to some extent for building purposes. Tuffs, rhyolites, and other porous volcanic rocks, are used sparingly as light-weight building materials. Mica schists are used as abrasive stones, for lining kilns and furnaces, and also for building purposes. Diatomite, tripoli and pumice are shaped into refractory or filter blocks.

#### BOULDERS

Boulders are "rolling stones," masses of rock loosened from the parent ledge by natural processes. Usually they are plentiful where the bedrock is close to the surface. In the unglaciated regions of southern United States boulders have not been transported far; therefore, in limestone regions they consist of fragments of the underlying limestone and in granite regions they are of granite. In New England and other northern glaciated territory, however, the ice sheet may have carried them for many miles, and in any locality may be found boulders of granite, limestones, sandstones or conglomerates assembled from widely scattered sources.

Boulders were used by the most ancient races for building primitive dwellings and they are still important construction materials. Local

boulders are used extensively in building rustic fireplaces, chimneys, basements, or entire houses. Many large mansions, clubhouses and mountain resorts are made of them. As they are obtained locally and are not a product of the quarry industry we have no record of the extent of their use. They are used in making fences and retaining walls. Where boulders are abundant many miles of stone fences are a characteristic feature of the countryside.

#### ENDURANCE OF BUILDING STONE

Stone is one of the most enduring of all building materials. In the Old World many stone structures built 500 to 1000 years ago are still in good condition. There is great variation in the endurance of stones, however, and some of them decay comparatively rapidly. The conditions of outcrops that have been exposed for countless years are the best evidence as to resistance to weathering, though observations made on stone buildings are helpful; but in America few of our structures are more than 200 years old and most of them have stood for less than 100 years.

Tests of the enduring qualities of stone are now made in laboratories. By repeated freezing and thawing of wet samples the results of exposure for many winters and summers may be shown within a few weeks. Most commercial stones have been tested thoroughly and no reliable quarryman will recommend for exterior use any stone that will not pass the most rigid tests for endurance.

The question often is raised as to which kind of stone is the most enduring. A general answer cannot be given because one kind of stone may be susceptible to certain agencies of decay and quite resistant to other disintegrating forces. The carbonate rocks (limestones and marbles) are altered chiefly by chemical action and to a much smaller degree by physical agencies. The granites, syenites, and other igneous rocks, on the other hand, suffer most from physical agencies such as repeated expansion and contraction as a result of sudden and excessive changes in temperature. Sandstones with calcareous cement are affected in much the same way as limestones and marbles. Firmly cemented siliceous sandstones are very enduring. Slates are affected very little by solution, expansion or contraction, but a high calcium content may lead to rapid disintegration if the slates are exposed to acid fumes or solutions. Thus it may be seen that a marble or limestone might be less enduring than a granite in an acid atmosphere, and a granite might disintegrate more rapidly than a marble or limestone under extreme temperature changes. Even under adverse conditions, however, deterioration is an extremely slow process and any standard stone produced by reliable operators is so enduring that the question of permanence need scarcely be given attention.

The permanence of stone masonry depends not only upon the quality of the stone itself but upon quality of supplementary materials and workmanship. Roofs, gutters, flashings and window casements should be so constructed that water cannot run behind stone facing blocks. Masonry mortars of only the highest quality should be used. All bedded stones should invariably be placed in the wall with the bedding plane horizontal.

#### TESTS, SPECIFICATIONS AND MARKETING

The stone of no two quarries is exactly alike. To take advantage of this individuality in stone, architects and builders desire the utmost freedom in selection. Therefore specifications relate chiefly to workmanship and surface finish and little is said about color, texture or general appearances. The American Association of State Highway Officials, however, has issued specifications for ashlar and rubble to be used in bridges and incidental structures. They relate chiefly to soundness, durability and absence of seams, cracks or other defects.

Although few general specifications are in use, individual contracts may call for exacting requirements to satisfy special conditions. Thus, a patterned floor requires tile of different colors and it is essential that they should not differ greatly in resistance to abrasion. Stone for sanitary uses should have a very low ratio of absorption. Stone for laboratory use should be resistant to chemical action. These special requirements call for a great deal of testing and many results of tests have been published.<sup>15-17, 22, 23, 28</sup>

The simplest method of marketing dimension stone is to sell rough blocks directly to dealers or manufacturers, and a number of producers do this. Some have mills where they saw the blocks, and such producers may sell either slabs or rough blocks to dealers or manufacturers. Most of the large companies that produce structural stone have their own finishing mills. They take contracts for individual structures and fabricate according to specifications. Most monumental granite is sold in rough blocks to manufacturers of memorials, although some important quarrying units also manufacture. A large proportion of monumental marble is manufactured by the producing companies and is sold to the retail monument trade. Paving stones and curbing are sold to contractors, municipalities, or dealers.

#### FOREIGN TRADE

Building and ornamental stone are imported into the United States in considerable quantities. Most of the imports are marble from Italy, Spain, France, Belgium, Portugal and England. Granite, chiefly for memorials, is imported from Finland, Sweden and Canada. Travertine

imports from the well-known deposits at Tivoli, near Rome, attain considerable magnitude at times. Onyx marble, of which there is very small production in the United States, is obtained from Lower California, Mexico, and the Province of San Luis, Argentina.

Exports of marble are much smaller than imports. Granite, limestone, and sandstone exports are not classified separately and therefore cannot be determined exactly, but they are very small. Canada is the chief consumer.

The U. S. Tariff Act of 1930 provided for an import duty of 60 per cent ad valorem on manufactured, and 25¢ per cubic foot on unmanufactured granite. The duty on marble and onyx in rough blocks was 65¢ a cubic foot and on sawed and dressed over 2 in. thick \$1 per cubic foot. Smaller sizes carried a duty of 8¢ to 13¢ a superficial foot, with an

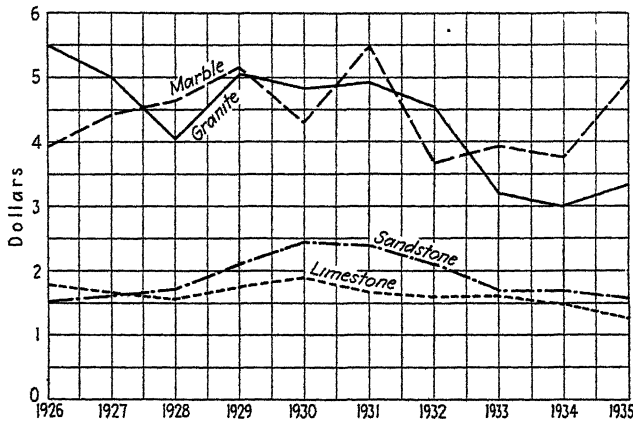


FIG. 3.—VALUE PER CUBIC FOOT OF BUILDING STONE (CUT, SAWED, AND FINISHED ONLY), 1926-1935.

additional charge of 3¢ if rubbed and 6¢ if polished. Manufactured articles chiefly or entirely of marble carried a duty of 50 per cent ad valorem. On unmanufactured sandstone and limestone the duty was 15¢ per cubic foot, and on hewn, dressed, or otherwise manufactured stone, 50 per cent ad valorem. Unmanufactured travertine carried a duty of 25¢ a cubic foot. The above rates of duty have been modified in some instances by reciprocal trade agreements.

#### PRICE HISTORY

Fig. 3 shows the average selling price of each of the major types of building stone from 1926 to 1935. The materials included are cut, sawed, and finished building stone only and the unit values are determined from quantities sold and the amount received f.o.b. mill as reported to the



United States Bureau of Mines. For the period covered, limestone and sandstone attained their highest unit value in 1930 and marble in 1931. Granite sold at a higher average value in 1926 than in any subsequent year. All varieties except sandstone show a decided drop in price since 1933.

Building stone probably suffered more than any other major industry during the depression, and the low prices during 1934 and 1935 generally indicate an effort to move material irrespective of profit in order to keep organizations together and to realize some income. A gradual strengthening of prices is to be expected when normal activity is approached.

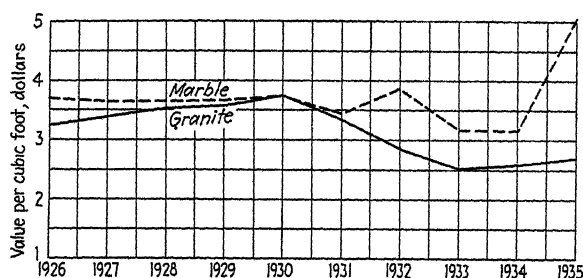


FIG. 4.—AVERAGE VALUE PER CUBIC FOOT OF MONUMENTAL GRANITE AND MARBLE, 1926-1935.

Fig. 4, based on statistics of the United States Bureau of Mines, shows trends in prices of monumental granite and marble for the same period. Granite prices increased gradually until 1930; thereafter they declined greatly until 1933 but recovered some of their losses in 1934 and 1935. Marble prices increased substantially in 1932. Although prices declined in some states in that year, in Vermont and Georgia, the states with the largest production, they gained more than 60¢ per cubic foot over the average of 1931.

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## CHAPTER XLIII

### CRUSHED AND BROKEN STONE

BY SEELY B. PATTERSON,\* MEMBER A.I.M.E.

**T**HOUGH stone has served as a building material since prehistoric times, its general use in the broken and crushed state is the development of little more than a century. The Romans used some small stone for a concrete made with lime and a considerable amount for the base of their great highways. Some hand-broken stone was used but most of the aggregate was gravel. It was early in the nineteenth century that McAdam and Telford, two Scottish engineers, successfully developed road building by the use of broken and graded stone, and with the development of crushing machinery for cheaper production, gave the great impetus to the use of crushed stone in highway work. Then came the increased use of Portland cement and the fabrication of monolithic structures of concrete, requiring great quantities of cheap aggregate. These uses, together with the requirements of ballast for the railroad systems and flux stone and lime for the iron and steel and other metallurgical and chemical industries, have brought the production of crushed and broken stone, with its competitor, gravel, to a position in the mineral industry second only to that of mineral fuel.

It is the scope of this chapter to cover commercial crushed stone, metallurgical stone, and the miscellaneous uses not detailed in other chapters of this volume.

#### ORIGIN

Rocks of all three groups—igneous, sedimentary and metamorphic—and from all periods of geologic time are the raw materials for the crushed stone industry. The igneous rock formations are the most extensive though they occur more generally in rugged and less populous districts. In southern New England, New York, New Jersey and Pennsylvania, comparatively small outcrops furnish a large tonnage and the production of crushed stone from this class of rock is also important in the other Appalachian and the Pacific Coast States. The terms “traprock” and “granite” are used in their broadest sense in the crushed-stone industry; “traprock” generally including all dense, dark, and fine-grained igneous rocks such as basalt, diabase, andesite and rhyolite, and

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"granite" the coarser grained igneous rocks, particularly those of lighter color, such as true granite and syenite. Traps, in general, are stronger and tougher than the sedimentaries and highly valued for such uses as railroad ballast and road metal. Granites possess great strength and hardness but in toughness they average less than the traps.

The sedimentary is the most important class as a source of crushed and broken stone. Rocks of this class vary more in physical properties than those of other classes. Their properties are dependent upon the character of the rocks from which they have been derived, the character of the cementing material, and the pressure to which they have been subjected in their formative period.

Limestones vary from soft chalk that can be crushed between the fingers to highly indurated types that show crushing strengths as great as many traprocks. Though on the average they are not as strong and tough as the igneous rocks, the sounder limestones are sufficiently so for most uses. They provide by far the greater portion of crushed and broken stone, for they are generally available near the centers of industry, have a broader range of usefulness and are more cheaply broken and crushed.

Sandstones are used to a limited extent for aggregate and riprap. Large amounts of the less firmly cemented sandstones are crushed for the production of sand.

Of the metamorphic rocks, marble and quartzite are the most important to this industry. Marble furnishes a large tonnage for most of the general uses of crushed stone and for many of the commercial uses where lime is required. It is also used in the crushed form as a facing in synthetic dimension stone. Quartzite is used mostly as a source of silica in the manufacture of refractories and glass.

Other kinds of rock are used in the crushed-stone industry but they are of minor importance. The porous volcanic rocks, pumice and tuff, are used to a small extent as light-weight aggregate for insulation and weight reduction in partition walls bearing no loads.

## COMPOSITION

Specifications for chemical and metallurgical stone are given in the paragraphs on particular uses. The chemical analysis of stone intended for such uses as road metal, aggregate or railroad ballast is less important than the physical properties, yet certain elements detract from its value for some uses. More than one or two per cent of iron oxide is likely to cause staining where the rock is exposed to the air and subject to oxidation. Sulphur will form gypsum by combination with lime, and the formation of gypsum crystals in cracks and joints causes internal strains that result in rupture and chipping. Vegetable matter, often contained

in the darker limestones, may form acids that can dissolve limestone or cement, weakening the structure, or causing efflorescence. Efflorescence is the formation of salts on the surface, or in the pores by evaporation from solution.

#### PHYSICAL PROPERTIES AND TESTS

The physical properties of rock are most important to the crushed-stone industry. Much has been accomplished in standardizing the physical tests of stone intended for highway uses. Brief outlines of the more common of these are given here and they are generally acceptable for other than highway uses. Complete details are contained in publications listed in the bibliography.<sup>13, 26, 27</sup>

Texture is the appearance of a fresh surface as affected by the size, shape and relation of the crystals or particles constituting the rock, such as coarsely crystalline and glassy. Structure differs from texture in that it refers to the larger features of the rock mass, such as bedding and cleavage.

Color plays a minor role. For stone surfacing of composition roofing, the granules of red and green shales and slates are used. For synthetic dimension stone, granite and marble chips are used for their color and texture. For facing concrete block and tile, a pure white aggregate is desirable, not only for its own whiteness but because a pure white base gives truer colors with the various pigments. In mass concrete, color is unessential, yet retaining walls are more sightly with white aggregates free from stain-producing elements. White limestone is particularly suited for the runways of aviation fields, for better visibility in landing.

Hardness is the property which resists abrasion. The Dorry test indicates the relative hardness by grinding the specimen with a standard grinding medium. A core 25 mm. in diameter, drilled from the rock specimen, is held against a revolving steel disk upon which crushed quartz sand of 30 to 40 mesh is fed. After 1000 revolutions of the disk, the loss in weight ( $W$ ) is determined and the coefficient of hardness is expressed as  $20 - W/3$ .

Toughness is the resistance to fracture under impact. The Page impact test determines this quality. In this test a cylindrical core, 25 mm. in diameter by 25 mm. high, drilled from the specimen, is subjected to the impact of a 2-kg. hammer falling upon a 1-kg. plunger of which the spherical end rests upon the specimen. After each blow the height of fall is increased by 1 cm. The height in centimeters of the fall that breaks the specimen denotes the toughness.

The Deval abrasion test is a measure of combined hardness and toughness and has largely superseded the Dorry hardness test. In this

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<sup>13</sup> References are at the end of the chapter.

about 50 pieces of the hand-broken, dried specimen, as nearly as possible uniform in size and shape, weighing 5 kg. are placed in a cast-iron cylinder. This cylinder is mounted upon an axis inclined  $30^{\circ}$  to the axis of rotation, so that in revolving it throws the pieces of rock from end to end twice in each rotation. After 10,000 revolutions at the rate of 30 to 33 r.p.m., the sample is screened on a sieve with openings of 0.065 in. and the percentage of the amount passing is used to express the percentage of wear. The French coefficient is an arbitrary way of expressing the wear and is equivalent to 40 divided by the percentage of wear.

The Los Angeles rattler test is now being actively studied by a number of state highway departments and commercial laboratories. It seems more indicative of the service value of aggregates than the Deval abrasion test and is less affected by the size of sample. In all probability, this test will replace the Deval test. The device for this test is a cylindrical drum, 28 in. in diameter by 20 in. long, mounted on a horizontal shaft. A 4-in. shelf on the inside extends from end to end. A 5-kg. sample of  $\frac{1}{2}$  to 2-in. aggregate is charged into the cylinder together with 12 cast-iron spheres weighing approximately 5 kg. The drum is turned 30 to 33 r.p.m. and after 100 and 500 revolutions the amounts passing a 10-mesh sieve are determined and expressed in terms of the per cent of the sample.

Strength is the resistance to fracture under steady pressure. The test for this consists in subjecting a cylindrical core of the specimen, 2 in. high by 2 in. in diameter, to steadily increasing pressure in a compression testing machine until fracture occurs. The pressure causing fracture is reduced to pounds per square inch.

Soundness is the quality that resists disintegration from weathering. Tests for this quality are not entirely conclusive. This fact is recognized by the American Association of State Highway Officials in the introductory note to the standard method of soundness tests for coarse aggregates which reads in part:

It is not intended that the test shall be used as an arbitrary basis for rejection of the material without taking other factors into account; consideration should be given to the results obtained with the materials when exposed to actual weathering conditions.<sup>26</sup>

The quality of the mortar is a greater factor in the soundness of concrete than the soundness of the aggregate, for an impervious mortar will protect the aggregate and, conversely, a porous mortar will allow greater exposure of the aggregate to weathering conditions. For such uses as railroad ballast and filter beds, the stone is entirely exposed and its resistance to weathering is dependent solely upon its own soundness, but the various methods for testing do not show close agreement and the value of such tests is questionable. The real test is that of actual service.

Freezing and thawing are the natural agents of weathering, so the obvious test is one of subjecting the water-soaked sample to alternate freezing and thawing temperatures. Variations in the results of this test on the same sample are mostly due to difference in speed of freezing.

The sodium sulphate test simulates the freezing and thawing action by causing the crystallization of the salt within the pores of the rock. In this test the sample of crushed stone is screened over a series of standard sieves and a definite amount of each size is washed, dried and immersed in a saturated solution of sodium sulphate and kept at a constant temperature for 18 hr. Each portion is then dried at 105° to 110° C. then cooled to room temperature; the soaking and drying is repeated five times, unless a different number of cycles is specified. The specimens are then washed free of salt, dried and screened with the same sieves on which they were retained before the test. From the amount passing each sieve, the weighted average of the whole sample is calculated. Additional information concerning the portions of  $\frac{3}{4}$  in. or larger is noted as to the number of pieces showing disintegration, splitting, crumbling, cracking or flaking.

Magnesium sulphate may be used for this test in a like manner, but tests made with the two different salts are not to be compared one with the other, as widely different results are obtained.

Specific gravity is the general term used to express the relation of the weight of a substance to that of an equal volume of water. Several different kinds of specific gravity are recognized but as applied to crushed stone the terms "bulk specific gravity" and "apparent specific gravity" are most important. Bulk specific gravity is the ratio of the weight in air of a given volume (including all voids) of permeable material to the weight in air of an equal volume of distilled water. Apparent specific gravity is the ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) to the weight in air of the same volume of distilled water.

The present standard method for determining both bulk and apparent specific gravity is the wire-basket method. The sample of about 5 kg. is immersed in water for 24 hr., then surface-dried by the use of a towel and weighed (*B*). The sample is then placed in a wire basket and its weight in water is determined (*C*). Finally, the sample is dried to constant weight in an oven and weighed (*A*). The following formulas then apply:

$$\text{Bulk specific gravity} = \frac{A}{B - C}$$

$$\text{Apparent specific gravity} = \frac{A}{A - C}$$

$$\text{Percentage of absorption} = \frac{B - A}{A} \times 100$$



Cementing value is the property of the finer particles to adhere to each other when moistened and dried. The test is made by grinding the sample with sufficient water to make a paste, molding this into briquettes 25 mm. in diameter by 25 mm. high, drying the briquettes at 100° C. and then subjecting them to repeated blows of a 1-kg. hammer dropped 1 cm. The number of blows required to destroy the briquette is called the "cementing value," which varies from zero for pure quartz to several thousand for some clays. Limestones give high cementing values and it is this quality that makes limestone screenings desirable for the binder in water-bound macadam roads.

A general test for the behavior of road stone under traffic can be made on a circular testing track. In one particular type of track, the material to be tested is placed in an annular groove, 14 ft. mean diameter, 18 in. wide, with a curb 6 in. high. A wheel mounted upon a radial arm is run around the groove upon the sample. Any type of tire can be used on the wheel and the pressure varied by counterweights on the arm. Thus traffic conditions are simulated and, in addition to aggregates, binders and other materials for road building can be tested.

The effect of an aggregate upon the strength of concrete is tested by mixing a sample of the aggregate with standard sand, cement and other ingredients, in proportion as they are to be used in practice. The concrete mixture is molded into blocks or beams and allowed to set for definite periods, usually 7 and 28 days. The blocks are subjected to direct compression to determine the crushing strength. The beams are held fixed at one end by clamping to a solid support and act as cantilevers. The other end remains free and weights are added to a pan suspended from this end until rupture occurs. From this, the modulus of rupture is figured. As concrete road slabs act as beams, the latter is an important test for highway paving. Other forms of beam tests are also made.

Sieve testing is done by hand or on vibrating machines. For the larger sizes it has been quite general to use test sieves with round holes and write the specifications accordingly. In stone plants, the punched plate with round holes for screening is being generally replaced by square-mesh cloth or plate with square perforations. There is some difficulty, when using stock square-mesh cloth, in meeting specifications based on tests with round-hole sieves. So there is now a trend toward specifying sizes based on square openings. A square opening is most closely approximated by a round opening having a diameter midway between that of the inscribed and circumscribed circles. Thus the diameter of the round opening should be 1.21 times the side of the square opening.

For sizes below  $\frac{1}{4}$  in., sieves with square-mesh cloth are used. As the wire sizes may vary with different manufacturers, the mesh per linear inch should not be used unless referred to a standard. The Tyler standard has been in use a long time. It starts with the National Bureau of

Standards 200-mesh with an opening of 0.0029 in. and each successive opening is increased by the square root of 2. By skipping every other sieve size, the ratio of 2 to 1 is obtained. For closer sizing, Tyler sieves are made with intermediate openings bearing the ratio of the fourth root of 2. The U. S. Standard uses the same ratio but starts the series from the 1-mm. opening.

Sedimentation and turbidimeter methods are used to determine particle sizes below the sieve sizes.

The "fineness modulus" is an index number applied to aggregates and is determined by adding the cumulative percentages retained on the 100, 50, 30, 16, 8, 4,  $\frac{3}{8}$ ,  $\frac{3}{4}$ ,  $1\frac{1}{2}$  and 3-in. square opening sieves of the U. S. Standard and dividing the sum by 100.

A sieve test is not easily interpreted until plotted. There are many methods of plotting designed to show up particular features of sizing; for instance, the direct or noncumulative method shows up strikingly an excess or deficiency of any particular size. For general purposes the semilogarithmic method of plotting cumulative percentages is most satisfactory. Fig. 1 illustrates this method and compares the sieve test with the specifications. The ordinates show the cumulative percentages in arithmetical progression. The abscissas are spaced in proportion to the logarithms of the numbers representing the apertures in inches or millimeters. By plotting according to the apertures, sieve tests can be compared though different standards of sieves may have been used. For the curves shown, the sand test was made on Tyler standard sieves while the specifications are written in the U. S. Standard. Logarithmic scale plotting of sieve apertures gives equal spacing for equal ratios and uniform grading is represented by a straight line from maximum to minimum size.

#### USES AND SPECIFICATIONS

The principal uses for crushed stone are: riprap, concrete, road metal, railroad ballast, sewage filter beds, sand.

Important uses for limestone and marble are: flux in smelting, cement, lime, sugar manufacture, rock wool.

The chief uses for fine or pulverized limestone are: agricultural, in chemical industries, glass, filler, rock-dusting of coal mines.

*Riprap.*—Riprap consists of heavy irregular fragments of stone and is used for protection against erosion, as on breakwaters, dam spillways, and fills across swampy land. The dense and less soluble igneous rocks are best suited for this work when available, though large quantities of limestone are used. Riprap brings a low price and is generally obtained from quarries along water transportation.

*Concrete Aggregate and Road Metal.*—There are no standard specifications for concrete aggregate and road metal for use throughout the United

States. They vary with the available rock deposits, character of traffic and climatic conditions, but as the Federal and State governments are the largest consumers, their specifications may be considered as standards for the particular locality. As these are changed from time to time, the latest specifications of the highway departments of the states in which the products are to be marketed should be consulted in evaluating the fitness of a rock deposit.

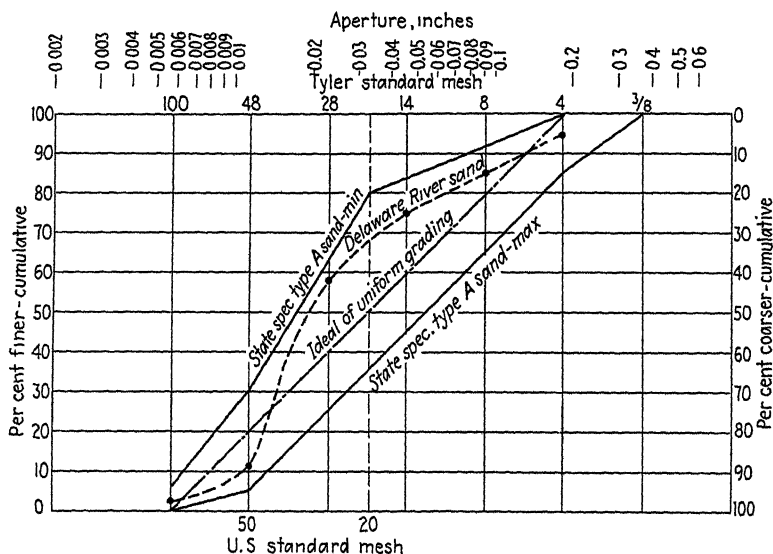


FIG. 1.—SEMILOGARITHMIC METHOD OF PLOTTING SIEVE TESTS.

Two principal qualities specified for highway use are wear and toughness. State specifications vary from 5 to 13 for the French coefficient, 10 to 17 for hardness, and 4 to 10 for toughness. Woolf<sup>33</sup> gives the following limiting test values.

TYPE OF CONSTRUCTION	PERCENTAGE OF WEAR NOT MORE THAN	TOUGHNESS NOT LESS THAN
Water-bound base course . . . . .	8	
Bituminous concrete base . . . . .	6	6
Concrete base course . . . . .	8	5
Water-bound macadam . . . . .	6	6
Bituminous macadam . . . . .	6	6
Bituminous concrete . . . . .	5	6
Concrete surface course . . . . .	6	6

Table 1 presents average values for the common rocks used in road building and a few of the rarer rocks occasionally used, as determined by the Bureau of Public Roads of the U. S. Department of Agriculture. The values given do not include all samples of each type of rock tested by the

Bureau but a sufficient number are represented to furnish values that may be considered as averages for the various rocks shown.

In designing concrete for greatest strength with the least cement, the tendency is toward a grading of aggregates producing the fewest voids, which is equivalent to greatest density. Specifications often require the furnishing of the coarse aggregate in a number of sizes to be recombined at the mixer to obtain greater uniformity. On large dams where coarse aggregate up to 6 in. is used, specifications may require this aggregate in as many as four sizes.

TABLE 1.—Average Physical Properties of Rock<sup>a</sup>

				Weight, Lb. per Cu. Ft.	Per Cent of Wear	Hard- ness	Tough- ness
I. Igneous	1. Intrusive (plutonic).	a. Granite . . .		167	4 3	18 3	11
		b. Syenite		171	3 3	18 3	15
		c. Diorite		179	3.0	18 2	17
		d. Gabbro		185	3.0	17.7	14
		e. Peridotite.		182	4.0	14 2	11
	2. Extrusive (volcanic).	a. Rhyolite..		159	3.7	18 3	19
		b. Trachyte		170	2.9	18.1	24
		c. Andesite.		166	3.9	17.0	18
		d. Basalt and Diabase		177 186	3.0 2.4	17.1 18.0	18 22
II. Sedimentary.	1. Calcareous	a. Limestone		165	5.0	14.1	9
		b. Dolomite		170	5.5	14.9	9
	2. Siliceous	a. Shale					
		b. Sandstone.		164	6.2	14 4	10
		c. Chert (flint)		159	9.4	18 2	12
III. Metamorphic	1. Foliated	a. Gneiss		172	4.9	17 4	10
		b. Schist		180	4.7	16.6	13
		c. Amphibolite		188	2.8	17 5	19
	2. Nonfoliated	a. Slate					
		b. Quartzite		169	3.2	18 8	18
		c. Eclogite		194	2 4	18 4	22
		d. Marble.		173	5 7	13 1	6

<sup>a</sup> From *Miscellaneous Publication* 76, U. S. Department of Agriculture, compiled by D. O. Woolf,<sup>33</sup> with which is combined the general classification of these rocks according to Dr. E. C. E. Lord, petrographer for the U. S. Bureau of Public Roads.

Goldbeck<sup>10</sup> lists the applicability of suggested standard sizes as shown in Table 2.

Specific gravity of the aggregate is important when it is used in dams or other structures where stability is a factor of design. Otherwise, the contractor usually favors the lightest stone that will pass specifications, for he is generally paid by the yard and buys aggregate by the ton.

TABLE 2.—*Applicability of Standard Sizes<sup>a</sup>*

Nominal Size Square Open- ing, in.	Circular Equivalent, in.	Uses of Different Sizes
$2\frac{1}{2}$ – $3\frac{1}{2}$	$3$ – $4\frac{1}{4}$	Water-bound macadam (soft stone)
$2$ – $3\frac{1}{2}$	$2\frac{1}{2}$ – $4\frac{1}{4}$	Water-bound macadam (soft stone) Bituminous macadam (soft stone)
$1\frac{1}{2}$ – $2\frac{1}{2}$	$1\frac{3}{4}$ – $3$	Bituminous macadam (hard stone) Railroad ballast (soft stone) combined with $\frac{3}{4}$ – $1\frac{1}{2}$
$1$ – $2$	$1\frac{1}{4}$ – $2\frac{1}{2}$	Water-bound macadam (hard stone) Bituminous macadam (hard stone) Special cold mixes Railroad ballast, combined with $\frac{3}{4}$ – $1\frac{1}{2}$
$\frac{3}{4}$ – $1\frac{1}{2}$	$\frac{7}{8}$ – $1\frac{3}{4}$	Choke stone for $2$ to $3\frac{1}{2}$ -in. stone, bituminous macadam Trickling filters Railroad ballast combined with $1$ – $2$ or $1\frac{1}{2}$ – $2\frac{1}{2}$
$\frac{1}{2}$ – $1$	$\frac{5}{8}$ – $1\frac{1}{4}$	Choke stone for $1\frac{1}{2}$ to $2\frac{1}{2}$ -in. stone, bituminous macadam Special cold bituminous mix Mixed in place bituminous surface Railroad ballast, combined with $1$ – $2$ (hard stone)
No. 4– $2$	$\frac{1}{4}$ – $2\frac{1}{2}$	Concrete highways Mass concrete Bituminous concrete
No. 4– $1\frac{1}{2}$	$\frac{1}{4}$ – $1\frac{3}{4}$	Concrete aggregate Bituminous concrete
No. 4– $1$	$\frac{1}{4}$ – $1\frac{1}{4}$	Reinforced concrete Unit concrete Traffic-bound roads Bituminous concrete Sheet-asphalt binder
No. 4– $\frac{3}{4}$	$\frac{1}{4}$ – $\frac{7}{8}$	Reinforced concrete Unit concrete Traffic-bound roads Coarse screenings for surface treatment Choke stone for $1$ to $2$ -in. stone, bituminous macadam Bituminous concrete, fine graded
No. 4– $\frac{1}{2}$	$\frac{1}{4}$ – $\frac{5}{8}$	Reinforced concrete Unit concrete Traffic-bound roads Fine screenings for surface treatment Bituminous concrete, fine graded Special cold bituminous mix
0–No. 4	$0$ – $\frac{1}{4}$	Fine aggregate Screenings for water-bound macadam

<sup>a</sup> Listed by Goldbeck.<sup>10</sup>

Some stone is not suitable for asphaltic concrete without special treatment, for it will not retain the coating of asphalt in the presence of water. Goldbeck says:

A number of tests have demonstrated that even the worst forms of hydrophilic rocks (those having higher affinity for water than asphalt) may be rendered immune to uncoating by giving them a pre-treatment of thin bituminous material which is then dried and hardened by the use of heat. Thin water-gas-tar is one of the materials which gives excellent results.<sup>11</sup>

Where fire-resistant qualities are to be considered, limestones rate high among the aggregates. The chemical action of breaking up the carbonate into the oxide and carbon dioxide absorbs heat and the calcium oxide formed upon the surface has low heat conductivity.

*Railroad Ballast.*—Standard specifications and methods of conducting tests have been proposed by the American Railway Engineers Association but limiting values are left largely to the individual roads. Many railroads specify only trap or granite for ballast, though this is a matter of available supply and relative costs. More limestone is used than any other type of rock. The breakage of ballast under the increasing size of rolling stock and from tamping not only allows displacement of the ties but it fills the voids and reduces its drainage value. Dusting reduces cleanliness of the road bed and if the dust has high cementing value it makes more difficult the reclaiming and cleaning of the ballast. The usual size required is  $\frac{3}{4}$  to  $2\frac{1}{2}$  in. Angularity of fragments increases stability or the resistance to displacement. Limiting values are placed on toughness, percentage of wear, soundness and absorption.

*Sewage Filter Beds.*—The sprinkling filter beds of sewage-treatment plants use large quantities of crushed stone of 1 to 2-in. size. The stone should be compact, should pass the sodium sulphate test for soundness, employing the test of 20 cycles, be free of sulphides, fines or clay, and the pieces sufficiently rough to furnish lodging places for bacteria. Limestone is highly satisfactory and silica not objectionable, if the stone is of uniform solubility and the silica evenly distributed.

*Sand.*—The fines produced in crushing make excellent sand for concreting, though it is not universally accepted. Because often the fines contain too much dust and dirt, there has developed a prejudice against their general use. Washing and classifying these fines will generally produce a product equal to natural sand and in many cases will give much greater strength tests in mortar than standard Ottawa sand. The sharp grains as compared to the more generally rounded grains of natural sand make the concrete somewhat more difficult to work, but care as to grading and the use of modern vibrating methods of concrete placement overcome this difficulty. In general, it must be sized uniformly and for the specified fineness modulus, and show 90 per cent of the strength of standard Ottawa sand when tested as a mortar with Portland cement.

*Flux Stone.*—Limestone is used in large quantities as a fluxing agent in the smelting and refining of iron and other metals to combine with the silica and alumina of the ores, fuel or metals, and form a readily fusible slag that is fluid enough at the normal temperature of the process to separate readily from the metals and flow from the furnace.

For the iron blast furnace, stone of  $\frac{1}{2}$  to 4 in. is used, though a closer sizing is preferred. With lump ores, a size similar to that to which the ore is prepared, usually 2 to 3 in., gives a more uniform mix in the charge. With fine ores, a size producing maximum porosity is desirable, to permit the easier passage of the gases in the furnace column. The stone should be strong and sound, to withstand the heavy burden of the high column of ore, and should withstand heat without breaking up. Some marbles decrepitate to powder under the heat of the furnace. Silica in the stone inflicts a double penalty. It not only reflects a smaller amount of the basic elements but reduces the available bases by the amount required to flux itself. Though blast furnaces can use stone with higher silica content than the open-hearth furnaces, and sometimes use a stone containing as much as 5 per cent silica, the market depends upon the available supply and relative costs, and rarely can a stone with over 1.5 per cent silica be sold for flux by commercial producers.

Other and important functions of the lime content of blast-furnace slags are: the reduction of loss of iron to the slag, control of the hearth temperature by varying the fusibility of the slag, and reduction of sulphur in the iron. The sulphur in the ore and fuel usually occurs as pyrites, which is reduced to FeS by heat. This FeS is readily dissolved by the metallic iron. To prevent this, the sulphur must be combined with an earthy metal that will join the slag to form salts. Calcium has a high affinity for sulphur and the reaction is more pronounced as the lime content of the slag increases, for then the calcium is less firmly bonded to other elements. Magnesium has less affinity for sulphur than calcium. The limitation of sulphur in the stone is about 0.5 per cent, but seldom does sound limestone contain as much as this. Phosphorus is also generally low enough, the limit for use with basic irons being about 0.1 per cent and with low-phosphorus irons about 0.01 per cent.

Theoretically, magnesia should lower the fusion point of slags, for double salts are more fusible than the salt of a single metal, though this is disputed in practice. Dr. Bowles states<sup>2</sup> that this "is possibly explained by the fact that slags consist of quite a number of components combined in complex ways, so that it is very difficult to determine just what would ensue from increasing a single element."

Specifications for flux stone for foundry cupola use are about the same as for furnace use, though the size is usually smaller, about  $1\frac{1}{2}$  to 2 in. High-calcium stone is generally desired, though sometimes dolomitic stone is used, and occasionally the foundryman likes more slag volume

and requests stone running several per cent in silica. Small foundries frequently find a sufficient supply of oyster shells in the immediate vicinity to fill their needs for flux.

In the basic open-hearth steel process, a high-calcium limestone, low in silica, sulphur and phosphorus, is required. High calcium is needed to reduce the phosphorus in the steel and carry it into the slag. Silica is much more detrimental than in blast-furnace flux, for the slags are more basic than blast-furnace slags and it reduces the available lime in a corresponding degree.

In copper and lead smelting, limestone is used as a flux when the ores are running more acid than basic. More often copper ores run basic and require silica for the fluxing agent, though as a rule siliceous ores can be obtained to provide a self-fluxing mix and obviate the necessity for buying barren silica.

*Cement.*—The raw materials for cement manufacture and the quarrying and preparation are covered in the chapter on cement. In general, the limestone purchased to raise the lime content of the raw mix must be high in calcium and low in magnesium.

*Lime.*—The quality of limestone for use in burning lime is dependent upon the use to which the finished lime is to be put. The manufacture of lime is an extensive industry, which is covered in another chapter.

*Sugar Manufacture.*—In the manufacture of sugar, lime is added to the juice of the cane or beets to neutralize the free acids. It is then precipitated with carbon dioxide gas and, in settling, it carries the colloidal matter with it and clarifies the liquor. As the manufacturer uses both lime and carbon dioxide, he usually buys the raw stone and burns it himself. High-calcium stone, about 97 per cent  $\text{CaCO}_3$ , free from impurities that would impart a taste, is required.

*Rock Wool.*—The use of limestones in the manufacture of rock wool is growing in importance. Details concerning the raw materials required, methods of manufacture, and the uses of rock wool are given in the chapter on insulating materials.

*Agricultural Limestone.*—Lime and limestone are used in agriculture to furnish the element calcium, necessary for plant life and to correct acidity of the soil. The use of lime for this purpose is covered in the chapter on Lime. Limestone is slower in action than lime and the calcium content is lower, yet the lower cost of the limestone often offsets these factors.

Some states specify the chemical requirements and the fineness of grinding for stone sold as agricultural limestone; others require only that the analysis be printed on the bag. High-calcium and magnesian limestones are used. The combined carbonates should exceed 95 per cent. It is marketed in various grades of fineness from 4 to 100-mesh. The meal as coarse as 10-mesh is not entirely dissolved the first year, so does



not give prompt effect, but if applied to the soil in greater quantity it will be as effective as the finer grade and last for a longer period.

*Chemical Industries.*—Raw limestone is marketed for many of the uses of the chemical industries listed in the chapter on Lime. Limestone fines are largely used to neutralize acid wastes. When added to the tanks, it is used in the pulverized form. In other instances, the acid solutions are run over a bed of limestone fines. These fines should be about  $\frac{1}{8}$  to  $\frac{1}{4}$  in., without dust, so as to permit free percolation of the solution.

*Glass.*—Limestone is one of the essential raw ingredients in the manufacture of flint, plate, window and bottle glass. For these kinds of glass, high-calcium limestone is required. Iron should not be over 0.3 per cent and for flint glass not over 0.03 per cent. Organic matter is limited to about 0.3 per cent. The size required is the same as that of the silica used, usually from 20 to 100-mesh, to allow an intimate mixture of the raw materials.

*Fillers.*—Most fillers are inert and are used to fill voids, add weight or impart color. Generally a product of 200 to 300-mesh or less is required.

For asphalt pavement, about 15 per cent of the mix consists of mineral filler. It hardens the product, gives it stability, and renders it less affected by heat. As the color is immaterial, pulverized stones of all kinds are used, as well as Portland cement, though limestone is most popular. Federal specifications call for limestone dust, dolomite dust, or Portland cement, passing 100 mesh with 65 to 100 per cent passing 200 mesh. Emery states<sup>7</sup> that the ideal filler is one that is graded from the largest to the smallest particle, so, that which is produced in the batch pulverizer may be better than the product of the air-swept mill, which latter is composed of particles approaching uniform size.

Pulverized limestone, marble, precipitated calcium carbonate, or chalk are generally used where a white filler is required. These are generally termed "whiting" though specifically this term applies only to pulverized chalk. Marble and chalk as a rule give a truer white product than limestone. "Each limestone deposit yields a whiting substitute with individual characteristics, the suitability of which for various uses can be determined only by actual tests."<sup>8</sup> Whiting is used for calcimine and cold-water paints, putty, as a component of glazes, enamels, and fluxed ceramic bodies, a filler in rubber for automobile tires, oilcloth and linoleum, in cigarette paper and as a coating on glazed paper, in white shoe dressing, white ink, dyes, and in toothpaste and many other articles.

*Rock Dusting.*—Rock dust is spread about the passageways and rooms of dry bituminous coal mines to reduce and prevent the spread of mine explosions. Dust for this purpose should be light colored, and free of carbonaceous matter and of free silica. Low silica content of the dust is necessary as a precaution against silicosis. A light color aids both in

judging the percentage of added dust in the mixture with coal dust and increases illumination in the mine. The rock should be ground to pass a 20-mesh screen, 50 per cent passing 200 mesh.

### PRODUCTION AND CONSUMPTION

The low price of crushed stone does not allow much expense in handling in and out of stock. The major portion is shipped as produced. Stock-

TABLE 3.—*Principal Uses of Crushed and Broken Stone Sold or Used by Producers in the United States, 1935-1936\**

Use	1935			1936		
	Short Tons	Value		Short Tons	Value	
		Total	Average		Total	Average
Concrete and road metal. . . . .	49,487,510	\$44,888,513	\$0 91	78,888,740	\$75,845,094	\$0 96
Railroad ballast. . . . .	5,267,010	4,011,469	0 76	7,891,080	5,992,693	0 76
Metallurgical. . . . .	12,191,660	7,902,717	0 65	17,724,880	11,576,156	0 65
Alkali works. . . . .	4,090,980	2,188,597	0 53	4,394,670	2,107,112	0 48
Riprap. . . . .	4,919,110	4,494,514	0 91	11,318,880	8,922,761	0 79
Agricultural. . . . .	2,140,370	2,656,728	1 24	3,743,710	4,406,703	1 18
Refractory (ganister, mica schist, dolomite, soapstone). . . . .	866,320	1,130,252	1 30	1,324,040	1,831,693	1 38
Asphalt filler. . . . .	152,040	363,163	2 39	210,370	498,031	2 37
Calcium carbide works. . . . .	237,340	135,844	0 47	348,170	178,694	0 51
Sugar factories. . . . .	460,460	640,375	1 39	540,470	754,967	1 40
Glass factories. . . . .	250,930	414,027	1 65	265,890	429,546	1 62
Paper mills. . . . .	188,090	339,372	1 80	255,880	399,861	1 56
Other uses. . . . .	1,396,330	3,141,863	2 25	2,046,120	4,430,932	2 17
Portland cement (including "cement rock") <sup>b</sup> . . . . .	81,698,150	72,307,434	0 89	128,952,900	117,374,293	0 91
Natural cement ("cement rock") <sup>b</sup> . . . . .	19,563,000	d		28,650,000	d	
Lime <sup>c</sup> . . . . .	5,974,000	d		7,570,000	d	
Total stone. . . . .	107,235,150	d		165,172,900	d	
Asphaltic stone. . . . .	814,109	1,449,406	4 61	547,333	2,420,792	4 42
Slate granules and flour. . . . .	226,510*	1,308,345*	5 99	289,650	1,646,780	5 69

\* From Mineral Market Reports, U. S. Bur. Mines, MMS 594, Aug. 25, 1937.

<sup>b</sup> Value reported as cement in the chapter on cement.

<sup>c</sup> Value reported as lime in chapter on lime.

<sup>d</sup> No value available for stone used in manufacture of cement and lime.

\* Revised figures.

piling is practiced not so much to meet future demands as to balance production against day to day shipments and because of the variation in demand for the different sizes. The total stocks carried are fairly uniform at all times so that figures of production may be considered equal to those of consumption.

Table 3 gives the production of crushed and broken stone in the United States for the different uses and Table 4 shows the production of crushed stone by kinds.

TABLE 4.—*Kinds of Crushed Stone Sold or Used by Producers in the United States in 1936<sup>a</sup> for Concrete, Roads and Railroad Ballast*

Kind	Concrete and Road Metal		Railroad Ballast		Total		
	Short Tons	Value	Short Tons	Value	Short Tons	Value	
						Total	Average
Granite . . .	6,835,130	\$ 7,295,702	1,110,410	\$ 890,795	7,945,540	\$ 8,186,497	\$1 03
Basalt and related rocks (trap rock). . .	12,484,900	11,852,160	839,590	843,553	13,274,490	12,695,713	0 96
Limestone . . .	49,308,570	45,808,424	5,058,530	3,602,649	54,362,150	49,411,073	0 91
Sandstone. . . .	3,983,700	5,475,916	387,860	291,131	4,371,560	5,767,047	1 32
Miscellaneous . .	6,331,440	5,412,892	494,640	364,565	6,826,080	5,777,457	0 85
Total. . . . .	78,888,740	75,845,094	7,891,080	5,992,693	86,779,820	81,837,787	
Average value per ton.		\$0 96		\$0 76		\$0 94	

<sup>a</sup> From Mineral Market Reports, U. S. Bur Mines, MMS 594, Aug. 25, 1937

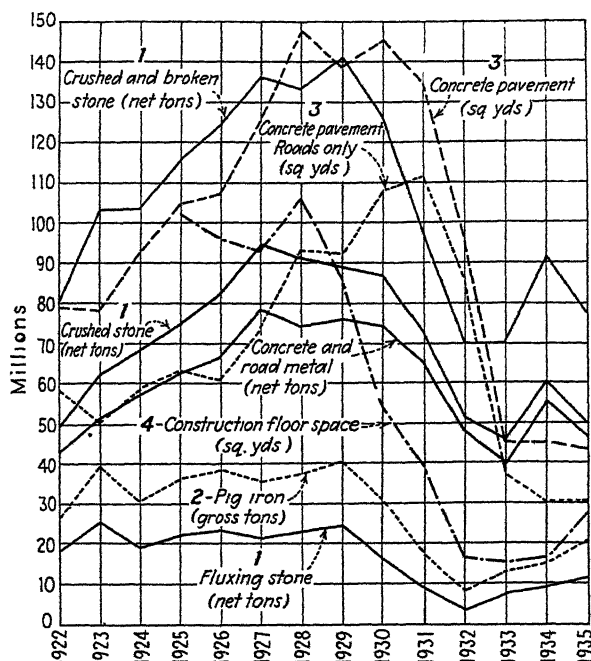
Fig. 2 shows graphically the trend in stone production since 1922 and compares it with concrete pavements and building construction; also, the production of fluxing stone compared to pig-iron production. As the market for any one quarry is restricted to a comparatively small area, national figures are not as helpful to the producer in judging his market as local figures. For graphs showing the long-time trend in the various states, see *Economics of Crushed-stone Production*, by Bowles.<sup>5</sup>

In Fig. 2, the production of crushed and broken stone for all uses closely parallels the curve of the square yards of floor space of private building construction, but the curves for crushed stone and for concrete aggregate and road metal do not drop as precipitously after 1929 because of the continuance of public road work at a higher rate than private building construction, to ease the unemployment situation. However, it did not help the commercial producer in the same ratio, as further figures show that the noncommercial production increased from 8.7 per cent of the whole in 1930 to 28.6 per cent in 1934, owing largely to the use of relief labor by governmental bodies in the production of crushed stone.

### PROSPECTING

Prospecting the deposit is essential to determine the quality, estimate the quantity, design the layout of pit and plant, and locate the disposal area for waste where it will not cover good stone. Success of the enterprise is dependent upon the thoroughness of this first step. Crushed stone is a low-priced commodity and profits are figured at a few cents per ton, so that there is but little margin for adverse errors. As with all natural resources, the more easily developed deposits are being depleted

and the less desirable ones must be used. The igneous deposits are more uniform in quality, by nature, usually stand out on higher ground, and are covered with less overburden, and surface sampling is less apt to mislead. Yet weathering may create inferior rock, and to determine the extent of this and the existence of faults or other major rock structures, drilling is advisable. Limestones in general are associated with low valleys and heavier overburden. They are less uniform and more distorted by folds



- 1- U.S. Bureau of Mines figures
- 2- American Iron and Steel Institute figures
- 3- Portland Cement Association figures
- 4- F.W. Dodge Corp. figures for residential and non residential construction in 37 Eastern States—public works and utilities construction is not included

FIG. 2.—STONE SOLD OR USED BY PRODUCERS IN THE UNITED STATES, 1922–1935, COMPARED WITH CONCRETE PAVING AND BUILDING CONSTRUCTION.

and faults, and rarely is there excuse for omission of thorough drilling before making expenditures for plant and equipment.

A map of the topography and location of roads and railroads, and a geologic sketch made from sampling the outcrops and plotting the dips and strikes is a first requirement. Information furnished in State and Federal geological reports should be studied and near-by quarries should be examined. If this report warrants the expenditure, drilling is the next step.

For prospect drilling, both the well drill (churn drill) and the core drill are used. The well drill does not recover pieces of rock large enough for

the physical tests, the most important for a source of stone. An experienced driller can get an idea of the hardness of rock from the sound and speed of drilling, but this is rather unsatisfactory. Where there are clay seams in the rock, the well-drill samples are not reliable for chemical tests, unless cased, for the clay washes into the hole and carries granules of silica and iron minerals that are not washed out and they contaminate the sample. Casing the hole the entire depth brings the cost close to that of core drilling.

The well drill provides a good means of testing the overburden. The hand augur is often used for this purpose but a small boulder will stop the augur and give incomplete results. The well drill can penetrate the boulders and determine definitely the top of the solid rock. The well drill also serves as a good auxiliary to the core drill. In pitched strata covered by considerable overburden, the general location of the desired formation can be determined, and with this information the core drill holes can be better located with a saving on this more expensive type of drilling.

In one type of core drill, shot of chilled iron or steel is fed under the bit with the wash water. The rounded shot break into pieces with sharp edges and cut the rock as it is carried around with the bit. The shot, being loose, will flow away into any large seam that may be encountered. Then the seam must be cemented. For such rock, the diamond drill is better suited, though occasionally the diamond drill may have difficulty entering a smooth surface at an acute angle after passing a seam, and cementing may be necessary.

In diamond drilling, the black, opaque, minutely crystalline carbonados (carbons) are better suited than the imperfect or colored crystal diamonds (bort), for their lack of cleavage makes them less liable to chip. Because of the increased cost of diamonds, setting the bit with a greater number of smaller stones is being practiced. Hard alloys may be substituted for diamonds in drilling the softer rocks. Tungsten carbide is the hardest of these alloys, having a hardness of 9 to  $9\frac{1}{2}$  on Mohs' scale as compared with a hardness of 10 for the diamond.

The entire core is not recoverable, owing to seams and the wear of sections of core upon one another. Even in solid rock free of parting seams, the core will break from the whipping of the drill rod. An average for the entire length of core recoverable is about 70 per cent of the depth of hole. When crushing part of the core for analysis or other tests, it should be split longitudinally with the device made for this purpose, leaving half for preservation and future reference.

The location of drill holes in massive formations or flat strata may well be at the corners of equal squares with sides 50 or 100 ft. or more, depending upon the uniformity of composition of the rock. For pitched strata, it is well to work on cross sections at right angles to the general strike. This means placing the holes closer together on the right angle than along

the strike. Well-drill holes must necessarily be vertical but diamond-drill holes can be drilled at any angle and should be as nearly as practicable perpendicular to the pitch of the strata.

Quantities are figured from the bulk specific gravity of the rock with allowances for seams of clay and open pockets. This allowance is a considerable factor in limestones.

Well-drill bits are commonly  $5\frac{5}{8}$  in. in diameter. Such holes cost from 40¢ per foot in sound limestone to \$2.50 per foot in traprock or granite. Diamond drills extract cores from  $\frac{3}{4}$ -in. to 3-in. diameter and the costs run from \$1.25 per foot for less siliceous limestone to \$6 per foot for the larger holes and harder rocks. Shot drills are built for holes up to 60-in. diameter, the larger sizes being used more for foundations and testing substrata for dams and bridge piers.

Geophysical methods for determining subsurface formations have been applied to the estimation of quarry overburden and the location of solid rock. Electrical resistivity and seismic methods have both been used. The former is more applicable to sand and gravel deposits. The Bureau of Public Roads has recently experimented with seismic methods and developed simpler apparatus of the refraction type suited to shallower determinations, which is of interest to the stone industry.<sup>24</sup> This method employs the principle of the difference in the rates of sound-wave transmission in soils (1000 to 6000 ft. per sec.) and in solid rocks (16,000 to 20,000 ft. per sec.). Sound waves are created at a point close to the surface by the explosion of a cap or small quantity of dynamite. Sensitive sound detectors are placed at various distances from the point of explosion or on the arc of a circle about this point. The moment of explosion and the waves received by the detectors are recorded upon a movie film driven at constant speed, which accurately indicates the time intervals. If the time interval for the first wave is less than would be required for the wave to be transmitted through soil alone, this wave will have traveled partly through rock, and by simple formulas the depth of soil through which the wave must have traveled to reach the rock is calculated.

### STRIPPING

There are few quarries fortunate enough to be free of the stripping problem. In a few instances, where the overburden is shallow and consists of friable soil, no attempt is made to strip, and the overburden is shot down with the stone and removed in the washing process.

*Hand Stripping.*—The removal of overburden by hand methods is seldom practiced today. Even in quarries employing hand labor for the loading of stone, the quarry operators are more than likely to give a contract for the stripping job to the operator of a portable excavator.

*Hydraulicking.*—No other method can compare with hydraulic stripping for low costs where the conditions are right, and it would seem ideal to thus wash the surface of the stone before loading and crushing, but seldom do the necessary conditions exist at quarry sites. Water under pressure of 90 to 200 lb. is directed through a nozzle (giant or monitor) against the bank to be moved. Additional bank water allowed to run along the face aids in carrying away the soil. Disintegration of the bank is accomplished both by direct impact of the stream from the nozzle and by the caving of the bank. Friable soil or unconsolidated sand and gravel cave readily when undercut. But the stream from the nozzle will bore holes in banks of tough clay without caving, and disintegration is a slow process of continuous action of the high-pressure water.

From 1500 to 2500 gal. of water is required for each yard of overburden moved. The slope should be away from the quarry face, otherwise the entire area must be stripped before quarrying operations commence. The slope must be steep enough to give the water in the sluice or channel sufficient velocity to carry the larger pebbles. Geike\* gives 40 ft. per minute as the velocity of water for moving coarse sand and up to 180 ft. per minute for pebbles of egg size. Boulders must be broken up and removed by hand.

A large spoil area is necessary to avoid stream pollution. If such an area is not available close at hand and at a lower level than the bank being moved, the debris can be carried to a sump and pumped by centrifugal dredging pump. The size of the pump is governed mostly by the size of the pebbles to be handled, which limits its use to the larger operations. Other disadvantages are that it requires special equipment not adaptable to other quarry operations and cannot be carried on in freezing weather. Developed in the placer-mining field, this method is now extensively used in the phosphate fields and in building hydraulic-fill dams. It has been used at a number of quarries, usually in areas of rough topography. For quarry stripping, hydraulicking costs are from 5¢ to 20¢ per cubic yard.

*Power Shovels.*—Power shovels are generally used for machine stripping. Costs are low and the same shovels can be used for loading stone in the quarry. Frequently the stripping can be done during the winter or other idle period of quarry operation and thus make unnecessary the purchase of any additional equipment. They can dig tough clay and inferior rock. One disadvantage is that they cannot clean out seams and in many limestone quarries the rock surface is very rough, solution cavities filled with clay extending to considerable depths.

Equipped with dragline boom and bucket, the power shovel can strip irregular surfaces and clean out vertical seams that are wide enough to

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\* Peele: *Mining Engineers' Handbook*, 782. New York, 1918. John Wiley and Sons.

admit the bucket. The machine equipped with dragline is slower in its movements of filling the bucket and swinging than the same machine with shovel dipper, and its capacity is thus cut about in half. Where the overburden is deep, it pays to do the stripping in two separate operations; first, a level cut just clearing the peaks of the rock with shovel equipped with dipper, and the remainder with dragline equipment.

*Power Scrapers.*—Various types of power scrapers are used for stripping but are limited to use in overburden loose enough to be plowed, and to short hauls. They do not clean up well where the rock surface is irregular. Such special equipment is not usable in other quarry operations, except the slackline excavator, which might serve for stockpiling of crushed stone. The roll-over type of power scraper is built in sizes up to 6 yd. capacity and the carrying type up to 24 yd. The slackline and drag scrapers may be used where available dumping area is at the side of the quarry but they lack flexibility.

*Transportation.*—Tractors with trailers are suitable for the shorter hauls of waste to the dump, trucks for the short and intermediate hauls, and locomotives with cars for the intermediate and long hauls. For conditions of deep overburden and long haul, locomotives with contractor-type side-dump cars give lowest costs as a general rule. Here track shifting is not so frequent. If dragline equipment is used the number of track changes at the stripping site is about one-half that required for shovel equipment, for the dragline has a reach of about twice that of its equivalent in the dipper type of shovel. Track-shifting machines are obtainable in standard and narrow gauge for use on the dump. When using trucks it is well to provide a tractor with scraper (bulldozer) for trimming the dump and maintaining the road.

Belt conveyers are used occasionally to convey the waste to the dump. The size of the conveyer is dictated by the size of the lumps or boulders to be handled, so that it is applicable to larger operations or those handling friable soil free of large boulders.

Where the area to be stripped is narrow and the waste can be disposed of at the sides, overcasting with dragline is most economical. This operation is one of throwing the excavated material from the bucket as far to one side as can be reached without the use of transportation equipment. If the pit is too broad to do this in one operation, overcasting the same material several times to reach the disposal area is often warranted by the elimination of any transportation cost. This applies to handling shallow overburden, for where the overburden is deep the pile may become too high for the dragline to handle. Overcasting is also practical in quarries of thin horizontal beds where the waste may be disposed of in the area that has been quarried out. This is the method used extensively in the strip coal mines. A portable belt conveyer, carried along with the shovel



or dragline, will extend the latitude of operations but is limited by conditions noted in the preceding paragraph.

## QUARRYING

### QUARRY PLAN

There are two general types of quarries. The pit quarry is the type in which the stone is extracted from below the general level of the surrounding country and as a rule the stone is hoisted to the crushing and treatment plant. Limestone operations are often of this type because of the characteristics of limestone deposits underlying the valleys. The bank quarry is the type in which the stone is taken mostly from above the level of the adjacent country, where the rock can be shot down to about the level of the crusher and no hoisting is required. Quarries of the latter type are usually self-draining and have no pumping problem. They are generally associated with the igneous deposits and with limestones in rugged country.

In flat-bedded deposits, the quarry layout can usually be made to suit the plant location, highways, railroads or other topographic features. In pitching and narrow strata, the layout is more involved. The hoist or point of attack should preferably be at the midpoint along the strike to reduce the average haul. The quarry face should be across the strata, at right angles to the strike, for better breakage in blasting. Blasting a face lengthwise of the strata will cause a slippage on the bedding planes, creating an overhang on one side and a heavy toe on the other.

The slope of the bench should be upwards at about 0.5 to 1 per cent, to drain water from the loading face and back to a central sump. Also, this slope tends to equalize the hauling effort by favoring the loads.

High faces are common in bank quarries because of the desire to get the blasted stone down on the crusher level. In pit quarries, the height of face for each bench is governed by the size of shovel, character of rock, amount of water to be pumped, and the necessity for providing more working points to increase production. In seamy rock that does not break up well in the primary blast, the face should be lower, for where large blocks remain in the blasted material above the reach of the shovel, undercutting the pile is necessary to bring them down and delays result in waiting for block-holing and blasting after the blocks are exposed.

Pumping costs can be reduced considerably in pit quarries by developing the working levels at shorter intervals. For example, instead of developing a level with a high face, if the same depth were developed in two stages, the pumping head would be reduced by half the height of the high face of the first plan for the period of time required to quarry the stone lying above the first stage of the second plan.

## DRILLING AND BLASTING

The distance from the toe of the face to the line of drill holes for primary blasting is called the "burden" and the distance between the holes in the line is called the "spacing." Suggested burdens and spacings are given in handbooks on the use of explosives. Generally the drilling and blasting practice in any quarry has been developed by trying various burdens and spacings with the explosive likely to be adapted to the character of the rock until the combination is ascertained that gives at lowest cost the results that are sought.

There is a difference of opinion on the best size and spacing for drill holes. One group maintains that solid rock is a good conductor of the shattering force of an explosion and claims that the explosive should be concentrated at fewer points to give greater disruptive effect. This leads to the use of holes of larger diameter with greater burden and spacing. Another group believes in a greater distribution of the total explosive charge by the use of smaller holes with closer spacing and lighter burden. The operator must study the characteristics of the formation of his quarry and decide which of these systems is likely to be preferable.

In the softer rocks associated with open or clay-filled seams, too much explosive at one point will tend to pulverize the stone adjacent to the drill hole and the shattering force will be cushioned by the clay or air space, leaving many large blocks unbroken. The larger holes with wider spacing are more applicable to the hard, dense rocks such as trap.

As the purpose of drilling is to provide space within the body of rock for sufficient explosive, a comparison of drilling costs must be based upon the volume of the hole which is proportional to the square of the diameter. Chambering (springing) is often resorted to in hammer-drill holes to enlarge them for primary blasting, particularly when they are horizontal, or nearly so. It is rarely practiced in well-drill holes.

For drilling, the hammer drill may serve well in thin strata of limestone worked in one bench or worked upon several benches separated by well defined parting planes, for in these cases the rock surface can be more easily cleaned. But when there is broken rock on the benches for some depth, it must be cleaned off to start the holes or else casing through the broken material must be used, to prevent it from falling into the hole, and the expense of either is likely to offset the lesser cost of this type of drilling. This broken rock results from over-drilling the holes of the bench above, which is necessary to break the toe where natural parting planes do not exist at the bench level.

The piston drill is seldom used in quarry operations and has largely been replaced by the hammer drill employing hollow drill steel. The hammer drill is built in sizes large enough to bottom a hole 2 in. in diameter at a depth of 35 ft. The larger sizes of hammer drills for pri-

mary work are better mounted on wagons for ease in moving, setting up, and handling the long drill rods. Hammer drills for block-holing are built in sizes as light as 20 lb., though a 40-lb. machine is about the lightest suitable for quarry use.

During the past few years detachable bits have been greatly improved and their use is now becoming common.\* Their chief saving is effected in underground mining where the handling of drill steel between sharpening shop and working face is always a problem and expense. They are particularly suited to the small quarry where difficulty may be encountered in getting forged bits properly dressed and tempered. There is also less investment in steel than when carrying the larger quantity of different lengths necessary with forged bits.

The well drill is most commonly used in drilling for primary blasts. The standard machine employs a  $5\frac{5}{8}$ -in. bit; for the tougher rocks, and where greater spacing is practiced, 8-in. holes are now common and 10-in. holes are sometimes used. Drilling is carried 2 to 5 ft. below the bench level, depending upon the burden and character of the rock, to avoid unbroken toes. Too deep drilling is to be avoided, for it makes it difficult for the shovel to maintain a level floor and requires more casing in drilling the bench beneath. All holes should be drilled accurately to depth. If the surface is irregular, accurate determination of the depth can be made by the drill operator with a hand level, sighting upon an established gauge. A log of the holes should be kept for reference in loading the shot. Explosive placed in clay seams is wasted and such waste may be avoided by splitting the charge so that the explosive is placed in solid rock and stemming placed in clay or open seams.

Multiple rows are often used, sometimes getting better fragmentation, but the particular advantage results from the reduction of the number of blasts with attendant cessation of operations near the face while charging the holes. Drilling costs are increased, for it is general practice to reduce the burden on the second and subsequent rows of holes. Holes are usually staggered from row to row.

"Buffer" or "blanket shooting" are terms applied to making a blast before the prior one is entirely cleaned up. A bank of loose stone prevents scattering the rock out on the quarry floor and confines it, to afford better loading conditions for the shovel, reduces the labor of cleaning up the floor, and reduces track work where this type of hauling is used. Better fragmentation may result, though this is questionable.

"Snake-hole" blasting requires that the drilling be done after the shovel has cleaned out the prior shot, so that it is applicable to long faces where time is afforded to accomplish the drilling before the shovel returns

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\* McH. Mosier: Progress Report on Investigation of Detachable Rock-drill Bits. U. S. Bur. Mines *Inf. Circ.* 6877 (Feb. 1936). 9 pp

on the next cut. A modification of this method for use on higher faces is to shoot the snake holes and then set the drill upon the pile of blasted rock to drill the unbroken bank above.

Tunnel blasting (known also as gopher-hole or coyote-hole blasting) may be practiced upon high faces where the structure of the rock is favorable. Small tunnels are driven into the face at the floor level and laterals in both directions from the end of the tunnel parallel with the face. The explosive, placed in the laterals, breaks out a wedge-shaped section at the base and the portion of the rock above this is lifted slightly and then falls of its own weight. The structure of the rock must be such as to give good fragmentation in this fall. Such is the columnar structure usual to trap rocks. It is not applicable to flat-bedded rocks, where an overhang would remain, nor to badly faulted rock. Sometimes well-drill holes are put down part way from the top to be shot simultaneously with the tunnel blast, aiding in fragmentation and also helping to control the line of back breakage and prevent overhang.

A greater amount of ammonia dynamite, which derives most of its strength from ammonium nitrate, is used in quarry work than any other type of explosive. Its freezing point is lower, it is safer to handle, and is cheaper than straight nitroglycerin dynamite. Ammonium nitrate is soluble in water, therefore cannot be used in wet holes. Gelatin dynamite will resist water indefinitely and is used under water where the pressure is considerable. Ammonia gelatin is a recent development and becoming popular for quarry use. It is similar to straight gelatin dynamite but a portion of the nitroglycerin is replaced by ammonium nitrate. It is water-resistant to a limited extent and can be used in wet holes where the water is not so deep as to subject the explosive to considerable pressure.

Various strengths of high explosives used are: 20 to 30 per cent for block-holing, 30 to 40 per cent for shales and cement rocks, 40 to 60 per cent for limestones, and 60 to 75 per cent for traps and granites. Often an explosive of greater strength is placed in the bottom of deeper holes to give better breakage at the toe, where the burden is greatest.

Gelatin dynamite has the highest rate of detonation and is used for the tougher rocks. In limestones and softer rocks, less pulverizing action near the charge and more uniform fragmentation are secured by the use of lower velocities. Lower velocity is also secured with larger grain size of the ingredients of the explosive.

Other types of explosive used in quarry work are nitrostarch, liquid-oxygen explosive (LOX) and black powder. Black powder may be used in rock where less than a 40 per cent explosive is satisfactory. The powder is usually graded in grain size for such use, to secure maximum density. Cordeau-Bickford fuse is used as the detonator.

Liquid-oxygen explosive was developed in Europe during the World War for industrial use because of the difficulty of obtaining materials for other types of explosives and it is now being successfully used in quarry work. Granular carbon is packed in cotton bags and soaked in liquid oxygen immediately before being loaded into the holes. Stemming should be fine stone or sand, as the gas resulting from the evaporation of the liquid oxygen will blow out solid stemming. Because of this evaporation of the liquid oxygen from the cartridges, it is necessary, for effective results, that loading and firing the blast be performed within a definite period from the time the cartridges are removed from the soaking tanks. This same quality is a factor of safety, for in about 24 hr. after the holes are loaded the residue is inert. The carbon cartridges are soaked in the liquid oxygen long enough to absorb an excess over that required for maximum effectiveness, and thus the evaporation during the normal loading time does not detract from its strength. This amount of soaking is dependent upon the size and character of the cartridges and the time required for loading the shot.

Liquid-oxygen explosive completely detonated is equal to about 150 per cent of its weight in 40 per cent dynamite. It can be used in wet holes but the rate of evaporation is more rapid than in dry holes. Its advantages are that no explosive is stored, as it is prepared immediately before use, and there is no hazard in handling misfires after evaporation has reduced the material to inertness. It has the greatest advantages in open-pit operations large enough to justify the initial cost of the liquid-oxygen plant, where the blasting routine provides a fairly steady operation of this plant. It is not recommended for use underground or where holes less than 3 in. in diameter are used, because of the more rapid evaporation of the oxygen in the smaller cartridges.

Cordeau-Bickford detonating fuse is particularly suited to the shooting of well-drill holes, where less fuse is required, than for the greater number of hammer-drill holes necessary to break the same amount of stone. It is placed from top to bottom of each hole and the charge may be split at will to avoid the wastage of explosive in clay seams and pockets.

A new detonating fuse, Primacord, has recently been placed on the market and received by the industry with great favor. The detonating agent is pentaerythrite-tetranitrate and is enclosed in a nonmetallic casing. Advantages are: light weight, less likelihood of breakage, simplicity of making joints, and high velocity (22,500 ft. per second).

#### LOADING AND HAULAGE

*Hand Loading.*—Hand loading is still practiced where careful selection is necessary, and in small operations at some distance from better equipped plants, where a shorter haul offsets the difference in production

costs. The type of labor for this work is more difficult to secure each year. An average loader working by contract can load about 3 tons per hour. The cost for explosives is greatly increased by hand loading because it is necessary to blast the stone to sizes small enough to be handled.

*Power Shovel.*—Power shovels are indispensable in the average quarry and the smaller quarries are adopting them because of the difficulty of securing sufficient hand loaders and to eliminate delays due to weather. Shovels are now made with dippers from  $\frac{3}{8}$  to 32-yd. capacity. The largest sizes are built more for stripping operations where overcasting is possible and a long reach desirable. A shovel with capacity as great as the transportation system would seem adequate, but too small a shovel is uneconomical though its capacity may be as high as the plant is designed for. The uneconomical feature lies in the inability of the smaller shovels to handle the larger pieces of rock left in the primary blast, which causes excessive delays and the expense of secondary drilling and blasting. In loading stone that is well broken up in the primary blast, such as shale or cement rock, this is no particular problem, but in many limestone quarries with seams of clay the primary blast does not throw the stone down upon the floor as well and the shovel must pull it out of the bank. This is hard on the shovel frame and mechanism and the smaller shovels cannot do it without entailing heavy repair costs. In hard digging, reduction in repair costs warrant the use of a shovel with a dipper one size smaller than that for which it is designed. The popular sizes are the  $1\frac{1}{2}$  to 4-yd. shovels for larger quarries and  $\frac{3}{4}$  to  $1\frac{1}{4}$ -yd. for quarries up to 500 or 800 tons daily capacity. Larger shovels are slower in action and the loading capacity is not in proportion to the size of the dipper.

Air has been used for powering shovels in underground work but electricity has largely replaced it in this field. Steam still holds high favor because of its flexible power characteristics and real digging ability in tough work. The disadvantages of steam—boiler trouble and expense from hard water, necessary service for supplying coal and water, and extra labor for firemen—have led to increasing use of electric, gasoline and Diesel shovels. Diesel power is replacing gasoline power because of the increasingly high taxes on gasoline and because of the more favorable power characteristics of the Diesel engine. The Diesel, gasoline and single-motored electric shovels perform the various acts of the cycle through clutches, a source of considerable trouble in the past but now very much improved.

The Ward-Leonard system of electric-shovel operation eliminates clutches and cumbersome controls by driving each operation (hoist, crowd and swing) with individual direct-current motors. Each motor is electrically connected to its own generator and all three generators are

mounted on a single shaft driven by a motor powered from the line current. The control of each motor is accomplished through control of the small field current. In comparing the cost of electric current with other forms of power, it must be considered that in the majority of cases electric power is used in the crushing and treatment plant and any additional power would cost less than the average cost per kilowatt-hour on the usual sliding scale.

Haulage from the loading point to the crusher is usually the neck of the bottle of operations and requires careful planning. The use of mules and horses still proves economical in many hand-loading quarries. Locomotive haulage requires the handling of trains of cars as single units for economy, and making up trains from many working points is a time-consuming operation. The idle time of small autotrucks sometimes used in hand-loading operations also renders this method uneconomical, unless separate loading skips are employed so that truck time is concentrated on actual haulage.

Locomotives are generally used for the average haul but have been replaced to some extent by motor trucks. Track gauges from 36 in. to standard gauge with 45 to 60-lb. rail are used. The circular form of track is most economical where practicable. In this the track is laid paralleling the loading face and the trains of cars travel around in one direction. A spring switch at the entrance to the circle eliminates any switch throwing. There need be no delay at the shovel waiting for cars to be switched. One locomotive less may be used at each shovel by pulling in the empties, dropping them for the shovel to spot and load, and pushing out the loads. Automatic couplers should be used for the saving of time and for safety.

The disadvantages of the circular track are the inability of doing selective quarrying, and in narrow quarries the circular track makes primary blasting difficult, for the corners cannot be reached for cleaning up. In narrow veins and for selective quarrying spur tracks radiating like fingers from the main line are used. Here the larger shovels with longer booms and dipper sticks make possible the loading of more cars per train on the dead-ended track. Passing tracks placed close to the loading point save time in spotting a new train.

Cars are from 1 to 50 tons in capacity. As every car is another unit to handle and dump, economy results in using the largest unit compatible with the gauge, minimum radius of curvature, and other conditions. Self-dumping cars of the contractor type are used frequently but the doors do not permit free discharge of large rock and are expensive to maintain. They are more suited to handling dirt. The self-dumping rocker type cars with V-bodies and no doors are for small units only. Of greater capacity for the same length of track space occupied, greater stability, and lower maintenance costs are the various types in which the body, heavily reinforced by I-beams, rests directly on the truck frame and

dumps to one or both sides by raising the opposite side with cable or ram. The sturdiest car of greatest capacity is the solid type that is dumped by overturning in the rotary tippie. In some operations, large skip cars with automatic end dump are used. For hand loading, low cars are essential and simple boxes of 1 to 2 tons capacity, mounted directly upon the axles, are generally used.

Power-shovel loading is extremely severe on cars, because of the height of fall of large pieces of rock, and the cars must be strong. Heavy wood planking on bottom and sides gives needed cushioning effect but must be covered with steel plate. Antifriction bearings are common now on quarry cars and worth their added cost in low upkeep and reduction in rolling friction. It is preferable to place the bearings in the wheels, allowing each wheel to turn independently, because curves in quarry work are usually sharp and this method of application of the bearings cuts down the tractive effort on curves and reduces wear on wheel flanges.

Locomotives are powered by steam, gasoline, Diesel, or electricity. Gasoline locomotives are today most common, in sizes from 2 to 35 tons. With nearly every person able to drive an automobile, gasoline-locomotive operators are easy to obtain and repair shops for gasoline engines are everywhere. The Diesel-driven locomotive is gaining in favor. The most troublesome feature of the Diesel engine has been the injection valve and this difficulty increases as the size decreases. The design has been so improved that units for as small as the 8-ton locomotive are now giving satisfaction. Diesel-powered units are considerably higher in first cost than those of gasoline.

Electric drive for locomotives is very satisfactory, as are also the electrically driven burden-bearing cars, with or without trailers, one objection being the trouble in maintenance of third rail or trolley near the working face. Remote control of individually driven electric cars has the advantages of saving in labor and in having the car service under the direct control of one man, who can arrange to have each shovel best served with cars, which advantages in some instances will warrant the high first cost of such an installation. In this system, the third rail or trolley wire is sectionalized and each section is independently supplied with current, controlled from a central tower at a point where the whole operation can be observed.

Gravity may be used in hand-loading operations for dropping cars from the loading face to the main line or foot of incline and returned by animal or locomotive. Where the haul is short and the face high, so that track changes are less frequent, gravity may be used to advantage for handling cars in shovel loading. The empties are placed on one level, dropped by gravity to the loading point, and returned by gravity on a circular track to a lower level whence they are hoisted. Large cars are



desirable to reduce the number of units handled as the hoist cable must be carried by hand from the upper to the lower level.

Autotruck haulage has the features of mobility and flexibility, which harmonize with these same features on the modern shovel with crawling or walking traction. There are no derailments to tie up the whole operation. Attack at the loading face can be made at any point and be changed quickly to another when desired. Comparative costs are difficult to obtain, as these costs vary so greatly with size of equipment, length of haul, and general conditions. Each quarry is a study in itself. In general, the truck is more suited to the shorter haul. While the locomotive has been more or less standardized for a number of years, the truck has been subject to rapid development. Its load capacity has been greatly increased and it is now more rugged and better suited to the severe work in quarries. The Diesel engine, with its power characteristic of greater torque at lesser speed giving greater power for the harder pull, is especially suited to heavy truck duty. Good road bed is as important to truck haulage as it is to track haulage.

In pit quarries of a single level, the primary crusher might be placed in the pit and the crushed stone transported to the rest of the plant on the surface by belt conveyer, bucket elevator or skip. Motor-truck haulage costs increase rapidly with increased grades, so that if the crusher is at a higher level, it is generally more economical to dump into a skip at the working level. Skips can be hoisted in balance and can be operated automatically by push-button control, for they travel from fixed point to fixed point, whereas in hoisting and lowering the stone cars they are generally spotted at different points in the trains and cannot be hoisted in balance nor automatically.

Derricks and cableways are used for hoisting stone boxes out of deep pits. They are more used in the slate and dimension-stone industries though derricks are occasionally used in hand-loading quarries supplying lime kilns, to lift the boxes to the top of the kilns. In one instance, a moderate sized limestone quarry hoists shovel-loaded boxes by derrick to the surface where three of these boxes are placed on each specially built car frame, taken by railroad to the cement mill and dumped into the crusher at the mill.

### CRUSHING

*Primary Crushing.*—The Blake type of jaw crushers and the gyratory crushers are usually used for primary breaking. Roll-jaw crushers (single roll and baffle plate) and hammer mills are sometimes used as primary crushers, the hammer mill giving the greatest ratio of reduction, but both these types are more suited for cement rock or other soft and friable rock. Of the two types most commonly used, the gyratory has

the greater capacity for the size of opening, for it crushes continuously during the whole cycle while the jaw crusher crushes only during the closing half of the cycle. In the average quarry plant, the jaw crusher with large enough opening to take the shovel-loaded stone will have sufficient capacity so that the greater cost of the gyratory is not warranted. Repairs and maintenance of the jaw crusher are simpler. Both types can be obtained with steel frames.

Jaw crushers are built with openings up to 66 by 86 in. and gyratories up to 84 in. The size of the opening should be more than ample to pass pieces that can be carried by the dipper of the shovel, for often pieces of rock, though somewhat smaller than the opening of the crusher, may arch against each other and bridge across the mouth. A pan feeder reduces the chances of bridging, and when such bridging does occur the feed may be stopped to loosen the jam without having to dig out a lot of rock that otherwise would be piled above the jam. The use of a feeder thus permits the use of a slightly smaller crusher. It also gives a steady flow of material through the plant and reduces the fines, for the proportion of fines increases with choke feeding.

The ratio of reduction in the jaw and gyratory crushers should be less than 6 to 1, particularly for the harder rocks. Greater reduction than this will tend to push the stone upwards instead of against the fixed jaw or concave, and will increase stresses on the machine. Some pieces of the product may be several times the size of the discharge opening in one direction, depending upon the character of the rock. The gyratory has the reputation of giving a product closer to cubical shape than the jaw crushers, because it breaks the slabby pieces against a curved surface, though the use of corrugated jaw plates in the jaw crusher tends to give the same effect. Corrugated wearing plates on both swing and fixed jaw are common, though the use of smooth plates on one lessens the side thrust on the swing jaw.

A feeder under the crushers saves the belt from wear and cutting by falling stone and is quite necessary where there is clay in the material, for a fixed-plate chute steep enough to carry clay gives such a velocity to clean stone that the belt is cut severely. If a feeder is not installed, a grizzly chute that allows fines to drop through onto the belt first to form a pad to receive the larger pieces is desirable.

*Secondary Crushing.*—The gyratory crusher is the popular machine for crushing to intermediate size. The conventional jaw crusher for this size of product does not usually have sufficient capacity, although it enjoys greater flexibility as to setting. There are several makes of double-action jaw crushers that have two fixed jaws, one on each side, thus crushing during both directions of travel of the swing jaw.

The roll-jaw crusher, with its single corrugated or knobbed roll crushing against a fixed curved plate, finds its best application in the

production of intermediate sizes. Its capacity is high on rock of no greater crushing strength than limestone, its headroom is low and its product contains a minimum of fines.

*Fine Crushing.*—Various kinds of machines are manufactured for fine crushing. Many use the gyratory principle with variations in the shape of the crushing surfaces. As the space between the crushing surfaces becomes thinner as it extends through the crusher, the circumference must become larger, to provide sufficient space to prevent blocking of the stone in its natural flow. This has led to the design of bell heads, spherical heads, flattened cones and disks. Furthermore, in the gyratory with the crushing surfaces close to vertical, the capacity to pass the crushed product is governed largely by the force of gravity. With flattened heads, centrifugal force added to that of gravity increases the speed at which the stone passes and also, by preventing the choke effect, lessens the percentage of undesirable fines in the product. The ratio of reduction in the cone and spherical crushers is 8 or 9 to 1. Double-roll crushers have large capacity and low headroom and produce sizes down to about  $\frac{1}{2}$  in. The reduction ratio should not be more than 4 to 1.

In most of the machines mentioned above, springs allow the release of one crushing member to pass tramp iron, or if "packing" occurs owing to excess fines in the feed.

*Pulverizing.*—Fine grinders are of many types and the selection in each case is based upon the character of the material to be ground, the magnitude of operation, and the specifications to be met for the finished product.

Hammer mills are used for producing the smaller sizes from the less abrasive rocks where the proportion of fines need not be kept low. The swing-hammer mill in the smaller size may be considered a fine grinder, its application in that field being for the coarser meals, such as agricultural limestone of the coarser grades and poultry grit, where the tonnage is small and simplicity is an important factor. It is not adapted to the harder rocks such as granite and trap. The ball, rod and tube mills, the ball-bearing type, and the roller mills (edge-runners) are the types of pulverizers generally used.

Closed-circuit grinding is practiced where extreme fineness is required. It gives a product closer to a uniform size, which may or may not be desirable, dependent upon the use for which the finished product is intended. In batch grinding, production is lower and cost higher but the product can be more accurately controlled, and it is used for special purposes where specifications are rather rigid.

Rock dust is usually ground dry but for the coarser products, such as crusher sand and glass sand, the wet process is used. Drying is sometimes accomplished in the grinding mill when the moisture content of the feed is not high, by heating the air used in sweeping the mill.

A grindability testing machine is built as a small-scale ball-bearing type of pulverizer, with the pressure on the balls kept uniform by the use of counterweights. A sized sample of the material is ground for a definite number of revolutions of the machine and the product is screened. Judging the results on the basis of Rittinger's law, the amount of new surfaces produced may be approximated from the amount retained on each sieve and the mean opening of the sieves and this compared with a standard.

#### ELEVATING AND CONVEYING

The belt conveyer is the most satisfactory device for conveying material within the plant and for elevating where space permits. The maximum slope permissible is about 18° or 33.5 per cent, though this may be exceeded if continuously loaded with fine or graded material. Careful design of the manner of feeding the belt to drop the material lightly and in the direction of belt travel will permit of higher speeds and allow the use of narrower belts. The bucket elevator is common in stone plants and is generally used where ground space does not permit the use of belt conveyers. Wear and tear and chances of shutdown are greater with bucket elevators than with belt conveyers and they should be avoided in the design of plant if possible.

To avoid the difficulties and upkeep cost of bucket elevators where a steeply inclined conveyer is necessary, the automatically controlled skip hoist is sometimes used, though its first cost is high and its operation intermittent and it requires a bin and feeder to give steady flow of material from this point through the plant. Pan conveyers are used for conveying short distances and are particularly suited for lump material and for feeders. Numerous other types of conveyers—drag, screw, vibrating and pneumatic—are not often used in crushed-stone plants.

#### SCREENING

Revolving and trommel screens have certain advantages that suit them to the very small plant. They serve as distributors over the bins, affording simplicity of layout, and are cheap. Universally used in the crushed-stone plant of a few years ago, they are rapidly being superseded by vibrating screens. Today the revolving screens finds their principal application in the scalping of larger sizes preliminary to crushing or washing operations or at the end of, or as an integral part of, revolving scrubbers. They do not serve well for screening sizes less than  $\frac{1}{2}$  in., particularly when the material is damp, and it is preferable to keep holes in the main barrel not smaller than one inch, obtaining the required smaller sizes through jackets. As the effective opening of a screen is the pro-

jection of the hole upon a horizontal plane, the cylindrical form of the revolving screen gives full size of hole only on a small portion at the very bottom and is reduced to zero as the screening surface is turned to the vertical position. The result is that the floor space is large relative to the screening capacity.

Bar grizzlies are used for scalping where accurate sizing is not required. Rolled bars should be tapered to reduce blinding. Scrap rail with the flanges cut off with acetylene torch except at the point of fastening makes an ideal grizzly bar. With its large head and thin web, it gives little trouble from blinding. Manganese-steel bars are used for abrasive stone.

The live-roll grizzly is a development of the past 10 years and is highly satisfactory for screening the larger sizes. This consists of a sloping series of horizontal rolls, each roll grooved to give the screening size desired, allowing the undersize to fall through. Each roll is turned at a slightly greater speed than the preceding one to minimize the crushing action between rolls. Large capacity (for single-stage screening) per unit of floor space occupied, ability to handle large stone, and quiet operation are its advantages. Another feature is that in handling stone associated with clay where subsequent hand picking is practiced, the clay is rolled up into balls, which facilitates the picking operation. An objection to this type is the time required to change rolls (or disks upon the rolls) when changing the screening size.

The vibrating screen was used originally for very fine screening but is now developed for all sizes, being used in one instance at least for producing riprap. The size of material to be screened dictates the type, magnitude and frequency of vibration. The tendency is toward flatter screen surfaces for more thorough and more accurate sizing. Horizontal vibrating screens are now available. Vibration is imparted to the screen surface either by direct attachment to the screen cloth or by attachment to the frame carrying the cloth or plate. The objections to the direct attachment are the flexing and breaking of the cloth and the difference in magnitude of vibration at the point of attachment from that at the sides. More uniform action is obtained by vibrating the screen frame but this increases the difficulty of preventing the vibration of the supports and the building itself. Better design, suspending the whole mechanism on spring supports or using rubber cushions, and more precision in dynamic balancing, have reduced this fault.

The vibrating effect may be produced by an eccentric, by cams, or by electric solenoids. Vibrating screens are made with single, double, triple and quadruple decks. Triple and quadruple-deck screens are subject to corrective capacity factors on the lower decks owing to material dropping on them some distance from the top. In making a single separation into two sizes, and where the material runs through a comparatively long range in size, it is advisable to use the double-deck screen, using

the upper deck with heavier cloth or plate to protect the finer cloth on the lower deck from wear by the larger particles.

#### WASHING

Washing is a comparatively new problem to the crushed-stone industry but is becoming increasingly important as the cleaner deposits become exhausted and as the specifications are being written more rigidly. The simplest method of washing is that of using water jets upon the material during the act of screening. This is sufficient where the foreign material is a small amount of readily separated surface soil or other friable material. A jet with a round hole playing the water against a curved plate to give a fishtail spray is a simple device and eliminates the trouble from clogging of fine holes or thin apertures common in other types of sprays.

The scrubber consists of a cylindrical or conical shell fitted inside with lifters to agitate the material and to carry it forward. Chains are sometimes suspended inside the shell to increase the scrubbing action, particularly in treating smaller sizes. Restricted openings at the ends keep a certain level of water in the shell and the flow of the wash water may be counter to that of the stone or it may be discharged with the stone to be separated later on a screen. The dewatering and sizing screen may be integral with the scrubber or a separate device. The scrubber will remove a coating of clay or soil and break up the more friable material.

To break up tough clay, greater churning and grinding action is required. This is well provided in the log washer, a device used for many years with clay iron ore, manganese ore and phosphate, and recently applied to the washing of aggregate and flux stone. As now manufactured, it consists essentially of two built-up steel logs on which is mounted a series of paddles. The logs are fitted with shafts at the ends and turn in bearings outside the enclosing steel or wooden box. They are sloped at an angle of  $5^{\circ}$  to  $10^{\circ}$ . The feed is introduced into the trough near the lower end and the stone is moved up the incline and discharged out the front end by the propeller action of the paddles, which form interrupted screws turning in opposite rotation. The lifting and churning action of the blades cut and reduce the clay balls and the action of the stone, working piece against piece, further grinds and scours the clay so that the clay and finer particles of stone float off in suspension in a counter current of water. The logs turn at a low speed of 16 to 25 revolutions per minute.

Recent developments of conical and cylindrical scrubbers use the principle of the ball mill, allowing a greater load in the shell and turning at a greater speed, thus grinding the clay balls with the stone itself. Other recently developed machines for washing stone are those employing the combined principles of log washer and scrubber.

There are many devices for washing sand not especially suited to the washing of larger stone, though some of these are being used in the crushed-stone industry to recover stone sand.

### STOCKPILING

Storage bins usually are sufficient only for a day's run, though silos of large capacities are sometimes used. To provide for the variations in demand of different sizes, ground storage is commonly required. Segregation of sizes is to be avoided in stockpiling. In building stockpiles by dumping directly from auto trucks, dumping the stone in layers will minimize the segregation. From such a stockpile the material may be reclaimed by small shovel, bucket loader, or drag scraper.

For plants where ground storage is a large item, piles are usually built up by elevated belt conveyers with trippers to discharge the belt at any desired point. The carriage carrying the tripper may also carry shuttle conveyers at right angles to the main belt, to broaden the pile. Reclaiming is usually performed by conveyers in tunnels under the piles. Traveling cranes, excavators equipped with long booms and clamshell buckets, or drag scrapers may be used to clean up material that lies beyond the natural slope to the tunnel gate.

In the radial storage system, the stone is dumped from a fixed point and chuted to any of a number of compartments radiating from this central point. Shipment loading is from a single point beneath. A circular track about the piles may carry a traveling crane, or a traveling tower for a drag scraper, to serve for moving the material from the live-load space to dead storage and reclaiming it again when required.

A lowering ladder will reduce breakage of stone in bin or storage pile. This consists of a vertical chute filled with baffles at short intervals, breaking the fall of stone into many short cascades. It is well to provide a screen at the end of the reclaiming belt to remove any fines produced in stocking.

### PORTABLE PLANTS

High freight rates and the extension of roads and public works into virgin territory are the factors leading to the increased production by portable and semiportable plants for specific projects. Portable plants are better adapted to sand and gravel production, where large crushers are not necessary, but every year finds improvements in the light crushers so that today a 9 by 40-in. jaw crusher is available in portable plant equipment and considerably larger for the semiportable.

Portable plants are built upon their own running gear and semiportable plants are light to be transported on trailers and quickly set up

at the site. An advantage of this kind of plant, in addition to the saving in transportation costs, is that it can be moved from point to point of the deposit, with resultant selective quarrying. The disadvantages are higher costs of labor, drilling and explosives because of the smaller crushers used, the waste in sizes that cannot be used on the specific project, and the delays to the construction job caused by breakdowns of the stone plant unless large stockpiles are maintained. These points are too frequently overlooked by the contractor when planning the installment of his own plant. The commercial producer will furnish the exact quantity of sizes required with no waste to the contractor and will deliver at the peak demand necessary to keep the job progressing at maximum efficiency.

Because the quantity of material for such projects is not great, all too often portable plants are installed on the basis of superficial observation, without any work having been done to properly develop the deposit. Many sad experiences have taught that, even with portable plants, thorough knowledge of the deposit and conditions will save unnecessary expense and costly delays in the prosecution of the contract.

To meet this competition, many commercial producers are prepared to furnish and operate the portable plant for the contractor. With their experience and personnel, they can do this to the greatest satisfaction, and thus securing the tonnage, they can then decide whether or not it would pay them to furnish this crushed stone from the portable plant, or, by shipping from the commercial plant, offset some of the transportation costs against fixed costs.

#### UNDERGROUND MINING

Though crushed-stone production is usually associated with open-pit quarrying, the amount won by underground mining methods is increasing rapidly. Thoenen<sup>31</sup> reports 64 active limestone mines in 1925 and the production from this source in 1924 amounted to 5,838,000 tons. As deposits favorable to open-pit quarrying become less available, attention is being directed to those requiring underground mining methods. Conditions necessitating the turn to underground methods are heavy overburden and the working of deposits to greater depths. Advantages gained are the production of a cleaner stone, protection from the inclemencies of the weather with resultant steadier production, and, with some mining methods, the ability to use stopes for storage of broken stone.

As the product is one of low value, mining methods using a minimum of timbering are employed. Frequently the underground operations are started by driving tunnels from the open pit into flat-lying strata as the overburden becomes too heavy for economical removal. This develops into tunnel mining or breast stoping. The tunnel is fanned out to provide a broad working face (breast) and pillars of rock are left at irregular



intervals to support the roof. The size and spacing of the pillars is dependent upon the strength of the rock in the pillars, the pressure of the ground, and the character of the roof. Breast stoping consists of breaking the stone from the vertical face for the entire height in one operation. It is applicable to strata not over 10 to 12 ft. thick and those lying flat or nearly so.

The room-and-pillar system of mining differs from breast stoping only in the manner of developing along precise mathematical lines, leaving regular pillars evenly spaced. This system may also be used in slightly inclined strata, driving the rooms level along the strike of the deposit.

In flat strata thicker than 10 to 12 ft., a combination of the breast stope with underhand or back stope, or both of these, may be used. Here entry is gained by breast stoping, and from this entry the bottom or the back is drilled and broken. Or the back may be drilled from the top of broken rock, as in the shrinkage-stope system.

The glory-hole (milling) and the shrinkage-stope systems are used for thick deposits and steeply inclined strata. In the shrinkage-stope system, 60 to 70 per cent of the broken stone is left in the stope and cannot be drawn until the stope has been entirely completed, so development must proceed well in advance of shipping requirements, thus entailing considerable development expense; but it provides a large reserve of broken stone to meet irregularities in demand.

Many variations of these systems are used to meet the particular conditions in each case. Thoenen gives details of the various systems used in limestone mining.<sup>31</sup>

The caving system is employed at one limestone mine<sup>22</sup> where the rock is coarsely crystalline and hard, though brittle and considerably broken by faults and fractures, and somewhat fissured by minute water courses. In the system as used there, the stone is cut off from the solid by shrinkage stopes in blocks about 200 ft. on a side and undercut by the room-and-pillar system.

Thoenen<sup>31</sup> found the average costs delivered to the crusher for 48 limestone mines in the United States to be about \$0.964 per net ton. Averages do not help in deciding upon the economics of using underground methods in any particular instance. Each case must be decided from its own special conditions. The costs reported varied from \$0.57 to \$1.27 per ton, the lower costs being less than for many open-pit quarries and the higher costs often warranted by the place value of the deposit.

#### COSTS AND CAPITAL INVESTMENT

In a study of quarry costs, Thoenen has compiled the returns from questionnaires sent to quarry operators in 1927 and reports them in detail for limestone production<sup>23</sup> and for traprock, sandstone, and granite.<sup>29</sup>

Dr. Bowles<sup>5</sup> gives the following figures for the capital investment per annual ton of production at crushed-stone plants:

	INVEST- MENT PER TON
Average of 16 limestone plants each producing less than 100,000 tons per year	\$2 12
Average of 10 limestone plants each producing between 100,000 and 250,000 tons per year....	\$1 28
Average of 9 limestone plants each producing between 250,000 and 500,000 tons per year.....	1 48
Average of 5 limestone plants each producing between 500,000 and 1,000,000 tons per year....	1 21
Average of 8 limestone plants each producing over 1,000,000 tons per year	1 14
Average of 4 granite plants	1 20
Average of 12 traprock plants.	1 68
Average of all the above plants	1 25

These are depleted values representing the actual replacement values of the land, buildings and equipment, and new cost would be somewhat higher. The figures do not include financing, promotional or initial operating expenses. Production capacity in these figures are actual average production over a two-year period. For all the quarries reported, the investment in land and mineral rights amounts to \$0.19 and plant equipment to \$1.06 per annual ton capacity.

#### MARKETING

Marketing of crushed stone is essentially a local problem. Deposits are common and transportation costs relatively high, so that they possess place value, and the market must be near by, seldom exceeding 50 to 100 miles. Monopoly is rare and competition keen with prices close to costs. Prices of labor and the materials entering into the production costs have been fairly stable for a number of years so that stone prices have been fairly uniform, the variations being more dependent on locality.

For the past six years, private construction's demand from the aggregate industry has been at low ebb, though showing marked revival in 1935, and dependence of the industry has been proportionally more on roads and public works. Here specifications are fixed by governmental bodies with inspectors in the field having no leeway for personal judgment. Acceptance is general at the point of destination. Handling from stock to car and car to truck is conducive of segregation, and as sampling for sizing tests is difficult at best and transportation charges are often more than the price at the bin, great care must be exercised to prevent serious losses on rejected material. Most bidding requires a study of delivery, unloading and handling costs.

The crushed-stone demand is seasonal as a whole. Very little moves during the winter months except in the Southern and Pacific States.

On road work, smaller sizes are in greater demand in the early spring for patching the ravages of winter. Later in the spring, the surface-chipping and oiling programs are rushed, to be finished before the heavy tourist travel commences, and this also requires the smaller sizes. Larger sizes for concrete move in greater quantity later in the season, after grading has been completed and in the rush to complete the concreting before freezing weather arrives.

Flux stone is by nature a product that moves more regularly, and because it is less generally available it moves greater distances. The market is confined to steel and smelting centers. It is not entirely accidental that there are good fluxing limestone deposits near the steel centers, for limestone is a considerable tonnage factor and a good supply of this raw material has been a big element in the location of steel industries. One ton of pig iron requires about  $\frac{4}{10}$  ton of limestone for the blast furnace, and further treatment requires additional amounts in the open hearth and cupola. Flux stone is sold by the gross ton of 2240 lb. whereas crushed stone is sold by the net ton of 2000 lb. or occasionally by the cubic yard.

#### MODERN TRENDS

In the mining field, notable achievements have been recognized in the profitable extraction of the low-grade ores. The crushed-stone industry is one dealing entirely with low-grade material with narrow margins of profit, and in this extensive field lie opportunities for engineers. In the careful analysis of the problem beforehand, good design, and the use of the most suitable equipment, lie the economies that reduce costs so that a profit can be gleaned in this highly competitive business. Success is dependent upon the knowledge of all the facts, having them in proper form for easy and correct interpretation. Changes are so rapid today that past experience alone is not always the best guide.

There has been a trend toward larger plants for more complete mechanization, but increasing freight rates and improvements in machines have checked this. Large-scale production may cover many errors but size of plant does not always mean low costs that can absorb so much of the delivery differential. It is the neat balance between size of market within reasonable delivery cost and the most modern and suitable equipment well coordinated with good design that brings success to the crushed-stone plant, however modest. And it is the modest-sized plant that requires the most careful engineering to produce the economies without excessive first cost.

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## CHAPTER XLIV

### STRONTIUM MINERALS

By B. N. MOORE,\* MEMBER A.I.M.E.

**S**TRONTIUM is an essential constituent of celestite (strontium sulphate), of the rarer strontianite (strontium carbonate), and of a few very rare minerals. Celestite is the chief ore, but strontianite is much more valued because of its ease of manufacture. The public knows strontium mainly as the source of the red flame in fireworks, but it has many other chemical uses.

#### PROPERTIES

Celestite of commerce is a massive mineral, coarsely crystalline, fibrous, bladed, very fine-grained or earthy in texture. The color varies greatly with the nature of the impurities. Distinctive characteristics are insolubility in acids, high density (3.95 to 3.97) and crimson strontium flame. Strontianite of commerce is a massive mineral, more or less coarsely crystalline. The color varies with the impurities. Distinctive characteristics are effervescence with acids, density (3.68 to 3.71), and crimson strontium flame. At 18° C., 100 parts of water dissolves 0.0011 part of strontianite and 0.0114 part of celestite. The solubility of celestite is greatly increased by the presence of chlorides. Strontianite is more soluble in hot water than in cold, unlike calcium carbonate, and is slightly more soluble than the latter in waters saturated with carbon dioxide. At 100° C., 100 parts of water saturated with carbon dioxide dissolves 0.12 part of strontium carbonate and only 0.10 part of calcium carbonate.

#### ORIGIN AND MODE OF OCCURRENCE

According to Clarke,<sup>3</sup> strontium forms about 0.02 per cent of the crust of the earth and is present in most igneous rocks, but in smaller quantities than barium. In some rocks strontia (SrO) may amount to as much as 0.18 per cent.<sup>5</sup> There is also concentration of strontium in hydrothermal solutions, as shown by the increase in the ratio of strontium to calcium over that in the rocks from which they have been derived.<sup>10</sup> Strontium is the fifth most abundant metallic ion in sea water and occurs

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\* U. S. Geological Survey, Washington, D. C.

<sup>3</sup> References are at the end of the chapter.

to the extent of about 13 to 13.5 mg. per liter, or about in the ratio of 1 part to 30 parts of calcium,<sup>13</sup> but sea water is only about one-fourth saturated with respect to strontium.<sup>13</sup> The skeletons of some marine invertebrates consist essentially of strontianite, and many shells contain strontium in the same ratio to calcium as does sea water.<sup>13</sup> Marine limestones and salines contain small amounts of celestite, which are concentrated by circulating waters into small veinlets and concretions.

Under certain conditions large masses of celestite are deposited from sea water with anhydrite. The presence of large amounts of celestite in some saline deposits and its absence in others suggest that the deposition of celestite depends on whether certain as yet undetermined concentrations of sulphate and chloride ions are reached in the brines. The most extensive of these sedimentary deposits are those of the Keuper (Triassic) in England, of the Zechstein (Permian) in Germany, and of various Cretaceous formations in Texas, Arkansas and Utah.

The celestite deposits of Sicily are associated with the sulphur deposits. Spezia, elaborating earlier hypotheses, demonstrated the presence of notable amounts of strontium in the lavas of Sicily and in the deposits of active solfataras. He suggested that the celestite and sulphur were both deposited with the enclosing sediments and were derived from submarine solfataras.

Of the deposits of southeastern California and western Arizona those at Argos, Aguila and Gila Bend consist of large amounts of relatively pure celestite interbedded with and grading into tuffs. The strontium was probably derived from solfataras. Deposits in the Avawatz Mountains associated with gypsum and manganese may have been formed by the precipitation of solfataric emanations in saline lake waters.

Strontianite is widely distributed but generally in small amounts as the weathering product of celestite, and is generally regarded as a secondary mineral. Becker<sup>2</sup> regards the extensive deposits in Westphalia as having been precipitated with calcite from bicarbonate waters, but he does not specify whether these were ascending or descending waters.

#### DISTRIBUTION OF DEPOSITS

*United States.*—Veinlets and masses representing concentration by circulating waters are common in some limestones of the states east of the Mississippi River, and in southwestern Michigan and northeastern Ohio, where such masses are abundant enough to be separated by hand sorting in some quarries. The most noted of these was the crystal cave of Put-In-Bay, Ohio, from which 250 tons of celestite were obtained. Deposits of probable sedimentary origin have been noticed in Burnet,

Lampasas, and Travis Counties, Texas, where the Glen Rose (Lower Cretaceous) limestone locally contains from 1 to 10 per cent of celestite as nodules and lenticular masses.<sup>4</sup> Similar deposits are found in the DeQueen limestone member of the Trinity (Lower Cretaceous) near Martha, Howard County, Ark.

Celestite is associated with gypsum and manganese in late Upper Jurassic rocks in Emery and Grand Counties, Utah. It has also been found in some metal-bearing veins in Utah. The largest deposits, however, are those of Washington, California and Arizona.

A deposit worked during the World War occurs 1 mile southwest of La Conner, Skagit County, Wash.<sup>6</sup> The celestite forms pod-shaped and lens-shaped bodies as much as 30 in. thick in a brecciated zone about 3 ft. wide in partly serpentinized dunite. Calcite, dolomite and limonite are also present, and the celestite is in part altered to strontianite. The deposit is of hydrothermal origin and is estimated to contain about 12,000 tons. A similar deposit is reported to occur at the mouth of Ruby Creek, on the Skagit River in Whatcom County.

A large deposit of celestite lies on the south slope of the Cady Mountains near Argos station, San Bernardino County, California.<sup>9</sup> The celestite is interbedded with shaly and clayey tuff in a zone about 300 ft. thick, which crops out for a distance of 4000 ft. The beds are as much as  $5\frac{1}{2}$  ft. thick and are formed of massive, finely crystalline celestite, locally with small amounts of silica. One of the analyses shows as much as 92.6 per cent  $\text{SrSO}_4$ . The eastern part of the deposit is extensively silicified, but croppings in the western part indicate a reserve of about 600,000 short tons. Celestite deposits associated with gypsum and manganese occur in the Avawatz Mountains, San Bernardino County, California.<sup>9</sup> At one place nearly vertical beds of celestite as much as  $3\frac{1}{2}$  ft. thick are interbedded with gypsum over a distance of about 1000 ft. The reserves are estimated at about 12,000 short tons. A bed of celestite about 10 ft. thick overlies a gypsum bed about 150 ft. thick in the Fish Mountains, Imperial County, California.<sup>9</sup> The celestite contains about 93.8 per cent  $\text{SrSO}_4$ . The reserves are about 10,000 short tons.

Strontianite occurs in clayey and shaly tuffs in the low hills about 10 miles northeast of Barstow, San Bernardino, Calif.<sup>9</sup> The strontianite forms concretionary masses in the tuffs and most of it is not of commercial value. Under the loose soil the tuffs are so hard that prospecting for additional bodies is prohibitively expensive.

A large deposit of celestite occurs in the Vulture Mountains, Maricopa County, Arizona,<sup>9</sup> 15 miles southeast of Aguila. The celestite forms beds as much as  $3\frac{1}{2}$  ft. thick in a series of shaly tuffs. It is more or less silicified. Only part of the district has been prospected. Surface crop-



pings and pits reveal reserves of about 180,000 short tons in the prospected part. Celestite forms steeply dipping beds in a series of sandy tufts about 15 miles south of Gila Bend and 3 miles east of Black Rock siding. The zone is reported to extend for a mile and for a distance of 750 ft. the main bed is 2 to 3 ft. thick. The reserves for this part of the zone are estimated at about 9000 short tons.

*Canada.*—Spence<sup>12</sup> has described in some detail deposits of celestite and baryto-celestite in Canada. Hydrothermal veins 1 to 3 ft. thick, with one or both walls in limestone, are known in Fitzroy Township, Carleton County; Loughborough Township, Frontenac County; Landsdowne Township, Leeds County; and Bagot Township, Renfrew County, Ontario. The Bagot deposit has been worked in an open pit and a small mill erected. Here the celestite contains 14 to 18 per cent of barium sulphate and forms a much broken vein 2 to 3 ft. thick in dolomite. The vein has been greatly disrupted during crushing of the dolomite. It is worked in an open pit, and about 30 per cent of the baryto-celestite can be recovered by hand methods. The rest is separated by milling.

Other deposits are known near Birch Island, 80 miles north of Kamloops, B. C., and in Cape Breton County, Nova Scotia.

*England.*<sup>11</sup>—Both strontianite and celestite are found at many localities in England and Scotland, but commercially important amounts of strontium minerals appear to be limited to the celestite deposits of Gloucestershire and Somerset. The celestite occurs as irregular masses and lenticular bodies associated with similar bodies of gypsum in the Keuper marl (Triassic) and in smaller quantity in the Keuper dolomitic conglomerate (Triassic). It is noteworthy that in Leicestershire and Newark commercially important bodies of gypsum are found at this same horizon. Celestite also fills clefths in the floor under the Keuper, and some of these fillings are as much as 3 ft. wide, 40 ft. long, and 20 to 30 ft. deep.

The deposits have been worked at various places in Somerset and Gloucestershire since 1874, but at present the workings are confined to a strip about 1800 ft. wide running from Yates as far south as Golden Valley, a distance of about 7 miles. About 400,000 tons has been produced. No data are available as to size of reserves.

*Germany.*—Celestite deposits of commercial interest as described by Bartling<sup>1</sup> occur at various places in Westphalia in the upper Zechstein (Permian). The best deposit is that near Obergembeck. It lies on a ridge, in two parts: On the north side of the ridge, celestite forms a strip from 33 to 99 ft. wide and 1500 ft. long, and on the south side another strip of about the same width and 660 ft. long, both in dolomite. On the crest of the ridge, in the underlying Kulm (lower Carboniferous), there are numerous scattered masses of celestite as much as a cubic yard in

size. The celestite contains as much as 94 per cent  $\text{SrSO}_4$ . The deposit was originally estimated to contain 50,000 metric tons but has been depleted by working. Near Giershagen, celestite forms small masses 1 to  $2\frac{1}{2}$  ft. thick and a larger body 3 to  $5\frac{1}{2}$  ft. thick underlying an area about 50 to 75 ft. wide and 1650 ft. long. The material is thinly banded, red and yellow, fine grained and cavernous, with some druses. The quantity present was estimated to be about 20,000 metric tons. The  $\text{SrSO}_4$  content in this deposit is more than 90 per cent. A body of bluish white and red celestite about 40 in. thick was discovered in the upper Zechstein (Permian), near Helmscheid, in 1902. It contains about 25 per cent silica, therefore has not been utilized.

The chief sources of German strontium in the past were the valuable strontianite deposits of the region about Münster, in Westphalia, which are described by Becker.<sup>2</sup> Numerous irregular veins cut the Upper Cretaceous marls of the region. Strontianite is irregularly distributed in these veins as lenticular masses as much as  $7\frac{1}{2}$  ft. thick, but usually not more than 10 in. Fillings show a bilateral structure, with calcite next the walls and strontianite, commonly banded, in the center. Pyrite occurs in small crystals on both calcite and strontianite and forms masses in the marl walls. It is commonly believed that the deposits do not persist to depths of more than about 65 ft., but Becker cites one set of workings that reached 312 ft. and believes that there is a large tonnage left in the district at lower levels than have yet been worked. The total production has been about 80,000 metric tons, but there are no estimates of reserves.

*Russia.*—Bärtling<sup>1</sup> mentions deposits of celestite similar to those of Sicily, in the Pinega River region of Archangel Province, Russia. The celestite is said to form large masses of blue color and excellent quality.

*Sicily.*—Large amounts of celestite are associated with the sulphur deposits of Sicily. The celestite is crystalline and forms large irregular masses as well as crystals, but has not been described in detail. Large deposits are found near Girgenti and Caltanissetta, but regular production has not yet been established, though at times it has been large enough to set the price for English celestite.

#### PRODUCTION AND PRICE HISTORY

According to Becker,<sup>2</sup> production from the Westphalian strontianite deposits began in 1840 and rose from 30 metric tons a year to 200 tons a year in 1875. With the introduction of processes using strontium hydrate for desaccharizing beet-sugar molasses, the production was stimulated and rose to a maximum of 7883 metric tons in 1884. The demand for strontium salts led to production from the English deposits in 1875, although these had been known since 1799. By 1884 English celestite

had obtained a dominant position, and from that time to the World War the total production of 5,000 to 20,000 tons a year was exported to Germany for manufacture. Most of it was used in the German beet-sugar industry, and the rest was exported as finished salts, mainly the nitrate. During this period the production of strontianite declined steadily and after the war it averaged only about 100 tons a year until about 1930, when it began to rise. Since the war there has been some production of celestite in Germany, but data are not available.

The price of English celestite at the quarry<sup>11</sup> was £2 a ton in 1884. In 1885 it decreased to 5 shillings a ton, but in the period 1893 to 1903 it rose to £1. From 1903 to 1907 it decreased to 15 shillings a ton. In 1915 the price at the Bristol dock was about 14 shillings 2 pence (\$3.45) a long ton, and in 1933-1934 about \$7 a short ton, or about \$10 a short ton delivered at Atlantic Coast ports.<sup>8</sup>

The price of German strontianite<sup>2</sup> was about 400 marks a ton in 1876 but has fluctuated around 150 gold marks a ton in recent years. The price is limited by the cost of manufacture of competitive carbonate from celestite, and does not rise more than 5 to 10 per cent above the cost of manufacture of the artificial mineral.

Up to the time of the World War the United States imported sufficient strontium salts from Germany to meet the needs of the pyrotechnic, chemical and drug industries. When this trade was shut off, celestite was imported from England until the embargo of 1917, which greatly stimulated domestic production. With the entry of the United States into the war the embargo was lifted, but domestic production of celestite for 1917 was about 4000 tons. Since the war the domestic demand for strontium salts has been met by imports of German salts and English celestite, which is manufactured on the Atlantic Coast. The total amount of salts and ore used is from 1000 to 3000 tons a year—too little to encourage domestic production in the United States to compete with the pure, cheap, and easily obtainable English celestite. However, there is sufficient reserve of domestic material to prevent any shortage caused by possible embargoes.

#### MINING AND PREPARATION FOR MARKET

The production of strontium minerals has always been carried on in a small way and by simple methods. The English deposits are prospected by sounding in the soil with long rods. The hard masses thus located are mined in pits, and may be found to be either celestite or gypsum. Most of the workings are less than 20 ft. deep, but where masses are found in clefts in the underlying formations workings may be deeper. The celestite lumps are washed and trimmed by hand.

During the war the deposits in the United States were worked on a small scale and the celestite was hand-sorted. Their larger scale mining

need not be expensive, although beneficiation would be necessary before much of the material could be marketed under present conditions.

#### SPECIFICATIONS

The  $\text{SrSO}_4$  content of marketable celestite should be 90 per cent or greater, and the  $\text{SrCO}_3$  content of strontianite should be more than 80 per cent. Because of the availability of the pure and cheap English celestite, the minimum standard of 90 per cent  $\text{SrSO}_4$  will probably be continued for a long time. Much of the domestic celestite needs beneficiation to meet such a standard.

#### MARKETING AND USES

The market for strontium minerals is limited by the uses for its salts, and new uses are needed before the market can be expanded. The greatest demand in the past came from the German beet-sugar industry, which used strontium hydrate in desaccharizing beet-sugar molasses. The constant demand from fireworks manufacturers for strontium nitrate is based on the characteristic red flame of strontium, for which no other salts can be substituted. This use makes it both a peace time necessity because of its use by railroads in flares and a wartime necessity because of the military use of red flares and rockets. Small amounts are used by the chemical and pharmaceutical trades, and some of the metal is used in copper in small amounts to prevent blowholes in castings. Strontium chloride is used as an absorbent in a gas refrigerator<sup>8</sup> and celestite has been proposed as a substitute for barite as a filler in rubber goods.<sup>8</sup> The carbonate can be used as a flux and desulphurizer in steel furnaces.<sup>8</sup>

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## CHAPTER XLV

### SULPHUR AND PYRITES

By W. T. LUNDY,\* MEMBER A.I.M.E.

**T**HE forms in which sulphur is commonly found—native sulphur, sulphides of many metals and sulphates—are widely distributed throughout the world, and the two first mentioned are the principal sources of sulphur. Substantial amounts of sulphur in some form are recovered from gases from industrial plants, and from gases from smelters treating other minerals. Such sources of by-product sulphur are not covered in this chapter, which is confined to native sulphur deposits of Texas and Louisiana; those of Italy; other scattered deposits of native sulphur; and also to pyrites deposits found in many countries of the world.

Sulphur, a nonmetallic element, is found characteristically in well developed bipyramidal or tabular crystals of the orthorhombic system, as well as in stalactitic and earthy masses. The mineral has a distinctive yellow color, which may be dark and discolored by impurities; the streak is white; the luster is resinous; and the fracture is conchoidal to uneven. Sulphur is translucent to opaque; has a hardness of 1.5 to 2.5; the specific gravity is 2.05; and the atomic weight is 32.06.<sup>13</sup> The molecular weight of the solid is expressed by the symbol  $S_8$ , and that of the liquid and vapor ranges from  $S_2$  to  $S_8$ , depending on the temperature. Sulphur melts at a temperature between 234° and 248° F., depending on its crystalline state, to a liquid of dark amber color, the boiling point of which is 832° F. The viscosity near the melting point is about that of water and increases to 50,000 times that of water at a temperature of 380° F., subsequently falling rapidly to a low figure at the boiling point. Above 205° F. crystallization changes slowly from rhombic to monoclinic, and in cooling the transformation is reversed. The mineral is a very poor conductor of heat and electricity; is insoluble in water and nearly all acids; and is soluble in cold carbon bisulphide and carbon tetrachloride. It ignites in air at a temperature of 478° F., burning with a distinctive blue flame with evolution of sulphur dioxide, which forms a convenient and convincing identification test.<sup>41</sup>

#### SALT-DOME SULPHUR DEPOSITS

*General.*—The sulphur deposits within the area bordering the Gulf of Mexico in Texas and Louisiana are of primary economic importance by

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<sup>13</sup> References are at the end of the chapter.

reason of their known and potential reserves, their productive capacity, the ability to market this basic commodity at a reasonable price, and also the unequaled purity of the product as mined. These are the commonly called salt-dome type<sup>24</sup> of deposits, and formations with which sulphur is associated may be described as submerged islands of rock lying at irregular intervals in a sea of unconsolidated sediments of great areal extent and unknown depth.\*

*Sediments.*—The territory in which these domes are found is characteristically low-lying and flat, with little topographical relief other than occasional mounds, which evidently are the result of salt-dome intrusion. The areas contiguous to the Gulf of Mexico, and extending irregular distances inland, are composed largely of sea marshes with numerous shallow bays, lagoons, lakes and bayous. Progressing farther inland the surface rises very gently by a series of inconspicuous terraces, probably former shore lines, and finally merges at its interior border into a low rolling terrain. Although drilling has not reached depths much greater than 10,000 ft., it is generally assumed that sedimentary formations are not less than 20,000 ft. thick., as illustrated in Fig. 1. Drilling indicates that these formations dip and thicken in a southerly direction toward the Gulf of Mexico and that they have a slight pitch in an easterly direction. It is evident that the sediments were built up by deposition from larger streams when and where they met encroaching waters of the Gulf of Mexico. Drilling has been so intensive during the last 10 years that geologists are now able to describe this process in great detail and apparently reach logical conclusions on the age of formations and methods of deposition.<sup>6,58</sup>

*Salt.*—The salt, which is the basic formation of all domes of the Gulf Coast, is an essentially dry, compact, and coarsely crystalline halite. Anhydrite is the principal accessory mineral and, occurring as scattered grains and small crystals, averages about 5 per cent of the salt mass. The top of the salt on some domes is rather flat, while others exhibit a domal or anticlinal shape. Salt domes have a circular outline with steeply dipping flanks, and those that differ from this form show an elliptical tendency in configuration. In a few instances salt lies within 100 ft. of the surface<sup>67</sup> and in contact with unconsolidated sediments, but usually it is immediately overlain with cap rock. Available evidence indicates that salt has intruded the sediments at great depths from mother beds of unknown geologic age. Weight of the sediments upon the salt is evidently the force that caused the intrusion. A factor that has been suggested as contributing to the present position is subsidence of sedimentary formations resulting from settlement of the basement series.<sup>5,14,43</sup>

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\* Publications of American Association of Petroleum Geologists contain numerous papers on Gulf Coast stratigraphy and salt-dome geology.

*Cap Rock.*—Cap-rock formations,<sup>9,21</sup> consisting of limestone, gypsum and anhydrite and underlain by salt, show individual characteristics in size, depth, thickness and configuration, as well as in the relative proportions of principal and accessory constituents.<sup>25</sup> Fig. 2 illustrates typical cross sections of sulphur-bearing cap rock. The mineralogical basis of this series of formations is a hard and dark gray anhydrite superimposed on and lying in contact with salt. Gypsum when present may be a dense gray variety or a porous white material containing lenses,

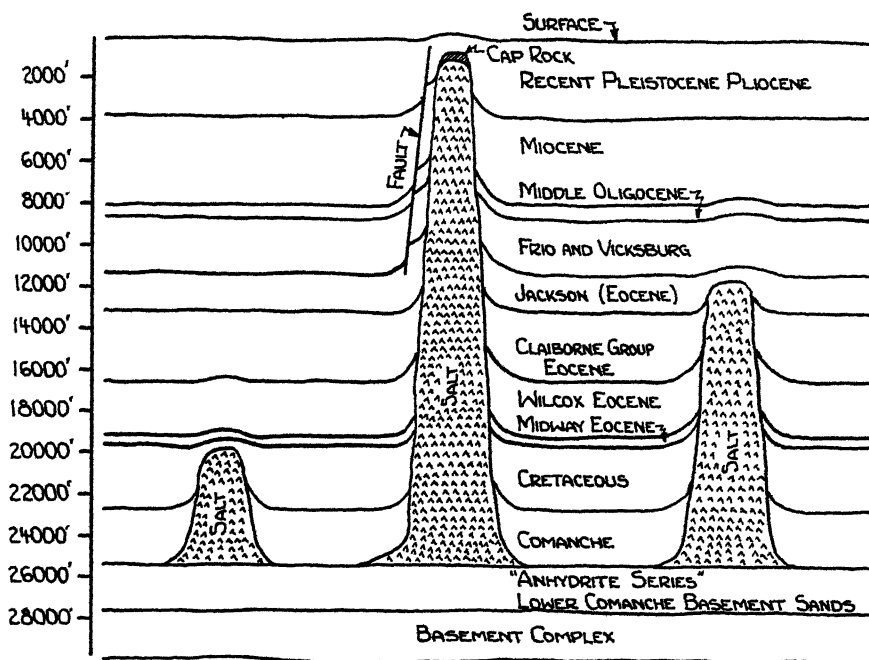


FIG. 1.—THEORETICAL CROSS SECTION OF PART OF LOUISIANA GULF COAST.

Showing depth to basement complex and types of salt plugs that may have affected various formations above 25,000 ft. (*Oil Weekly*, Jan. 20, 1936.)

nodules and well developed crystals of selenite. Limestone strata are relatively large in comparison to gypsum and anhydrite. It may be present as a single stratum, as a series of lenticular beds covering only a portion of gypsum and anhydrite, or in disseminated lenses and nodules included in the upper portion of the series. The limestone is typically a fine-grained gray carbonate interspersed with vugs, seams, fissures and cavities, which may be partly or wholly filled with calcite. Siliceous solutions have frequently indurated the limestone in the upper portions where it is typically barren of sulphur. Domes exhibit variations in thicknesses of cap rock from less than 50 ft. to more than 1000 ft. A universal constituent of cap rock is water highly charged with hydrogen



sulphide, polysulphides, sodium chloride and other salts, and having a temperature approximating 100° F.

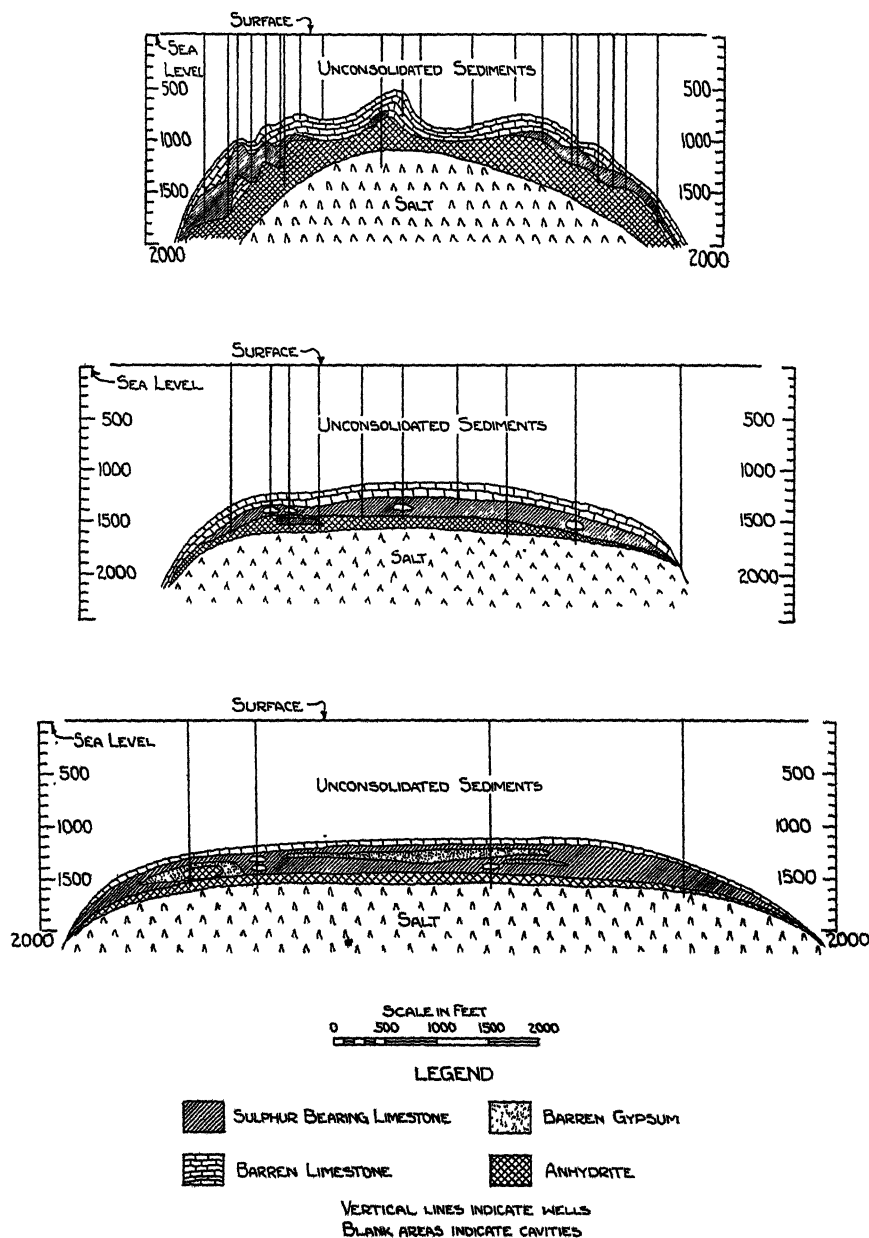


FIG. 2.—TYPICAL CROSS SECTIONS OF SULPHUR-BEARING CAP ROCK ON SOME SALT DOMES.

Current publications discuss two principal processes, with minor modifications, explaining the origin of cap rock, and both of these assume

the intrusive origin of the salt structure. One theory is that anhydrite was an original formation with and overlying the salt beds before inception of the movement and that it has been lifted to its present position with the salt.<sup>9,21</sup> In contrast is the theory that assumes that circulating waters dissolved vast quantities of salt during its upward movement. Its anhydrite content was thus concentrated as an insoluble residue on top of salt and was subsequently compacted and recrystallized, by the same forces causing the salt intrusion, into the hard and dense cap rock that lies as a mantle on nearly all salt domes.<sup>20,24,66</sup> Both theories assume that gypsum was formed by hydration of anhydrite. Structural and petrographic studies indicate that limestone, at least in part, is an alteration product of these formations. The amount of calcite present is very suggestive of the important part played by circulating waters.

*Sulphur Formations.*—Sulphur of mineralogic interest is quite persistent in many cap-rock formations, but deposits of commercial magnitude are found on only a small percentage of the known domes. The principal deposits are usually confined to the lower parts of the limestone and in some places substantial amounts extend into the gypsum. Sulphur occurs as well developed crystal aggregates in the seams and cavities of the porous limestone, and also in a semicrystalline or massive state as a filling in the openings of formations. The thickness of the barren cap rock varies from a minimum of 5 or 10 ft. to a maximum of 200 ft. The thickness of the sulphur horizon is seldom less than 25 ft. and may reach 300 ft. as a maximum, the average for bodies of commercial size approximating 100 ft. For a few feet, sulphur may be in practically pure state, but the average content of formations for the various deposits ranges from 20 to 40 per cent. These deposits show a marked difference in reserves, as illustrated by mines that have been worked to abandonment. With approximate cap-rock areas of 75, 800 and 2000 acres, respectively, for Sulphur, La., Bryanmound and Palangana, Texas, the production figures are generally reported as follows: Sulphur, 10,000,000 tons; Bryanmound, 5,000,000 tons; Palangana, 235,000 tons. The porosity of the sulphur-bearing formations has not been determined accurately, and it is generally assumed that it averages between 15 and 25 per cent.

*Distribution.*—The area in which coastal salt domes are found extends from slightly east of the Mississippi River to the vicinity of Corpus Christi, Texas, and inland from the Gulf of Mexico for approximately 75 miles. Within this territory, comprising nearly 45,000 square miles, the presence of 95 domes has been definitely established by drilling, and many other deeper structures have been indicated by seismograph surveys. Most proven structures have been prospected for sulphur, some more thoroughly than others, and deposits of commercial size have been mined from nine domes. These include Sulphur, La., by the Union

Sulphur Co.;<sup>33</sup> Bryanmound,<sup>34</sup> Hoskins Mound, Texas,<sup>39</sup> and Grande Ecaille, La., by Freeport Sulphur Co.; Gulf,<sup>72</sup> Newgulf\* and Long Point, Texas, by Texas Gulf Sulphur Co.; Palangana and Boling,\* Texas, by Duval Texas Sulphur Co.; and Jefferson Lake, La.,<sup>44</sup> by Jefferson Lake Oil Co. There are known salt domes or similar structures in regions other than the Gulf Coast of Texas and Louisiana, but there are no reported deposits of sulphur associated with any of them. Those best known include interior domes of Texas;<sup>49</sup> interior domes of Louisiana;<sup>59</sup> Rumanian salt domes;<sup>69</sup> German salt domes;<sup>61</sup> salt domes of the Isthmus of Tehuantepec;<sup>24</sup> Persian salt domes;<sup>26</sup> and salt structures in Colorado and Utah.<sup>27</sup>

*Prospecting.*—Since 1924 geophysical methods have played an important part in prospecting and locating structures favorable for sulphur deposits.<sup>56</sup> The normal procedure includes a seismograph survey of large areas having little or no surface indications of salt domes. When rock structure is indicated it is customary to supplement the seismograph with a torsion balance to secure more detailed information on size, depth and configuration of cap-rock area.<sup>4</sup> A few wells may then be drilled at scattered locations at distances ranging from 500 to 1000 ft. or more, and when sulphur is encountered in one or more wells a systematic drilling program is carried out.<sup>37</sup>

*Mining.*—The early history of the Frasch process at Sulphur, La., was covered most interestingly by Herman Frasch in an address upon receipt of the Perkin medal.<sup>18</sup> Since then a number of deposits have been found, developed, and a few abandoned because of depletion of deposits. Publications for the past 10 years have recorded comprehensively the methods and practices employed, and Fig. 3 illustrates typical well equipment as well as the principles of sulphur mining by the Frasch process.<sup>12, 45, 47, 51, 63, 73</sup> The following discussion, therefore, will be confined to topics of more general nature.

The Frasch process requires water in quantities, which may vary with the size of the plant from 1,000,000 to 10,000,000 gal. per day. The water, being heated from atmospheric temperature to at least 300° F., must be fairly pure originally and must be carefully treated to eliminate corrosion-forming and scale-forming constituents. Sulphur deposits of Texas and Louisiana have been generally fortunate in lying in close proximity to large rivers, which bring from inland areas water comparatively free of salt and other objectionable constituents. At some places well water containing natural sodium bicarbonate is mixed with river water for treating purposes. When mines lie close to the Gulf of Mexico the water occasionally becomes high in salt content, and large reservoirs are employed to store good water when available. Fuel to

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\* Located on Boling dome.

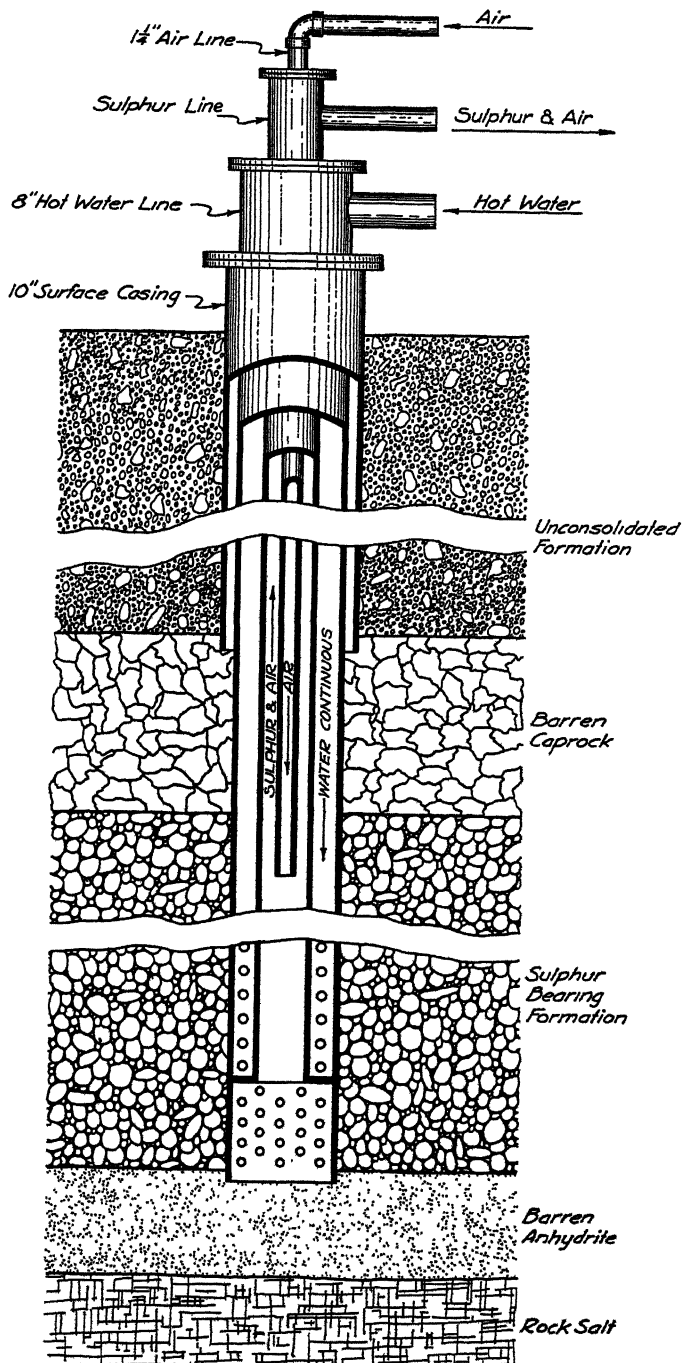


FIG. 3.—TYPICAL SULPHUR-WELL EQUIPMENT.

heat the water usually constitutes one of the largest items of expense, and a reliable supply at reasonable costs is an important consideration.

In equipping a sulphur property for production, probably the largest single item of cost is the central heating and power plant.<sup>46</sup> The first requisite for the plant is continuity of operations for an indefinite number of years, because interruption in supply of hot water to wells would endanger their loss by freezing of sulphur. The principal unit is boiler equipment to convert one-fourth of the water to steam, which is then mixed with the remaining three-fourths to raise the temperature of the whole to that required for mining purposes. The plant also furnishes steam for turbine-driven centrifugal pumps to transport water through thousands of feet of insulated pipe lines to wells and underground formations. It also has equipment for compressing air for raising the liquid sulphur to the surface, as well as electrical equipment to generate power. Electrically operated equipment is now used for a multiplicity of purposes such as drilling, lighting, pumping water and liquid sulphur, loading and transportation of sulphur, and operation of maintenance facilities including machine, blacksmith, carpenter and electric shops. Waste formation water, about equivalent in volume to that used for mining purposes, can be treated with plant stack gases to eliminate or greatly reduce the sulphide content before disposal.<sup>10</sup> Modern plants for sulphur mining have attained a comparatively high degree of efficiency. From 75 to 80 per cent of total heat in the fuel is put into the water by boiler equipment, and in addition, a substantial part of the heat in stack gases can be recovered.

The Bryanmound sulphur deposit was in operation from 1912 to 1935, and during this period four separate plants were constructed, not as replacements but as amplifications. At one period in its history Bryanmound, with an installed plant capacity of 23,000 boiler horsepower, delivered daily about 9,000,000 gal. of water for mining purposes with a fuel consumption of approximately 4500 bbl. of oil. The plants were constructed immediately over cap-rock areas and surface subsidence, due to extraction of sulphur, resulted in settlement of the two largest plants approximately 7 ft., without injury to any equipment. Subsequently plants at other domes have been located at safe distances from mining areas. The cap-rock area at Bryanmound comprises approximately 800 acres, and of this 296 acres was classed as commercially sulphur-bearing. Deposition of sulphur, characteristically spotty and irregular, was confined principally to the flanks of the dome. Substantial amounts were also found at irregular intervals over the entire dome area, principally in limestone but frequently in gypsum or in formations that changed with depth from limestone to gypsum. During the life of Bryanmound, 1897 wells were drilled, which resulted in a production of 2635 tons per well. There were critical periods when permanent sus-

pension seemed inevitable, but each time there was some change in conditions, such as improved prices, expanded markets and substantial reductions in fuel costs, which, together with more efficient methods, permitted continuation until 1935.

Formation conditions that should lead to efficient operation of the Frasch process include a thick and rich deposit of sulphur; absence of overlying barren cap rock, which retards or prevents subsidence of upper sedimentary formations into cavities; impervious strata above and below the sulphur deposit to prevent escape of hot water; and a uniform porosity to assist in an even distribution of hot water. As subsidence does not always take place naturally and quickly, a substitute method for filling the largest voids and channels in the rock structure has been developed. The desired effect is a more uniform porosity to increase efficiency in utilization of hot water. Mud is pumped into wells, at locations remote from mining areas, until the pressure reaches 200 or 300 lb. The amount of mud applied shows wide variations ranging from 25,000 to more than 300,000 cu. yd. per well. Innovations in methods of mud application to formations offer interesting avenues for improvements in mining sulphur.

Sulphur produced by the Frasch process is exceptionally free from foreign materials and usually requires no purification. One explanation for this fortunate characteristic may lie in the relatively greater wettability of gangue rock compared to sulphur in the presence of water. The result is that fine gangue rock when wet by hot water is either washed away from the vicinity of the well or floats on the surface of liquid sulphur and does not contaminate it. Mud, often consisting of large amounts of sand, gravel and shell, has been pumped into wells previous and subsequent to steaming, and in addition, the formations have a tendency to disintegrate as sulphur is extracted. The specific gravity of sulphur in the liquid phase is considerably less than either materials used in mudding operations or the formations. Notwithstanding these conditions, sulphur produced by the Frasch process is rarely contaminated and by analysis shows a very small ash content—usually less than 0.01 per cent. Infrequently organic impurities such as petroleum residues, soluble in sulphur, contaminate and discolor the product, and when this occurs quality and color can be improved by distillation or by adsorption.

#### ITALIAN SULPHUR DEPOSITS

*General.*—The sulphur deposits of Sicily can be listed among the outstanding mineral deposits of the world, due not only to their long history but also to known and potential reserves, as well as to the large number of people dependent on the industry for their existence. Unique methods of beneficiation developed under adverse conditions add further interest to the history of these deposits. Economically the Italian

sulphur industry<sup>68</sup> has encountered vicissitudes of many kinds, not the least of which was the reduction of its markets as a result of the development of the Frasch process in the United States.

*Formations.*—An ideal section of formations includes yellow, loosely cemented sandstone, argillaceous marls and blue clay, foraminiferous limestone interbedded with clay, and below this series of sediments the gypsum and sulphur-bearing formations. The entire region was subjected to severe faulting and folding which, together with erosion, complicated the regional geology and resulted in a rolling and semi-mountainous terrain. The sulphur-bearing member of these formations is composed of a series of strata of brownish cellular limestone interbedded with bituminous shales. The continuity is not equivalent to that of the overlying gypsum, but the sulphur-bearing formations lie in isolated basinlike deposits 1 to 5 miles long, from 1000 to 3000 ft. wide, which may reach a maximum of 200 ft. in thickness. Sulphur, in ore bodies of commercial size, is usually disseminated through the limestone, may lie in thin seams parallel to the bedding, or may occur as well developed crystals in the porous phases of the rock. As mined the grade of ore lies between 12 and 50 per cent, with an estimated average of 26 per cent.<sup>29</sup>

*Distribution.*—The region of sulphur deposition in Sicily is confined to the south central part, and the deposits are included in an area of approximately 495 square miles, constituting 5 per cent of the island. There are many mines within this area, and about 24 can be regarded as of primary importance because of their size, production and equipment. Relatively important deposits, similar to the Sicilian, occur on the mainland, and the principal mines are found in the eastern part of Italy. Although these mines are inferior to those of Sicily in some respects, yet the production has gradually encroached on that of Sicily until now they account for over 28 per cent of all sulphur produced by Italy.

*Mining.*—Because of the great number of years the Sicilian mines have been in operation, and also because of the size and extent of the deposits, methods of mining and beneficiation are the most highly developed for any deposits of this type in which the sulphur-bearing rock is brought to the surface through mine openings such as shafts, tunnels and drifts.<sup>55</sup> In recent years the largest mines have installed mechanical equipment for mining, tramming and hoisting. Development has been carried in depth to 800 ft., with workings extending great distances from the outcrops. Where beds worked are flat, the room-and-pillar system is used, and after pillars are robbed rooms are closed by waste filling or caving of the roof. The longitudinal cut-and-fill method of stoping is used where the dip is steep and this method is commonly employed in the larger mines. Development work is carried well ahead of extraction, and the mines can thus be worked efficiently and with high recovery.

As is true in all mines of this type, the danger of fires, especially in the richer deposits, is a serious hazard, and the presence of hydrogen sulphide in pockets in the formation is an ever present source of additional danger. The necessity for pumping large volumes of water constitutes a considerable item of expense in mining operations.

*Preparation.*—At one time the Calcaroni method was employed almost universally. This consists of carefully and skillfully constructed piles of ore 15 to 100 ft. in diameter and 3 to 15 ft. deep.<sup>54</sup> On being ignited, part of the sulphur is consumed and the heat of combustion liquefies the remainder, with recovery of about one-half of the sulphur. Inefficiency of the Calcaroni, together with large volumes of objectionable gases discharged into the atmosphere, led to development of the Gill regenerative furnace about 1880. As used at present, these furnaces consist of a series of masonry kilns, eight being the maximum, which are arranged in circular form and connected in series by flues. When filled with ore and ignited the products of combustion pass successively through the remaining charged kilns before being wasted to the atmosphere. Thus the gases and vapors transfer their heat to the ore; sulphur vapor is recovered by condensation; and relatively cool gases only are wasted. The cycle is continuous and extraction of sulphur approximates 80 to 85 per cent. As with the Calcaroni system, the product is contaminated with fine gangue material and analyzes from 90 to 95 per cent sulphur. Other methods for beneficiation have been tried, such as concentration by flotation, solvent extraction of sulphur, and steam application in closed containers.<sup>64</sup> To a very limited extent these methods have shown sufficient reduction in costs to displace the older systems.

#### MISCELLANEOUS SULPHUR DEPOSITS

No known areas other than those described above are characterized by numerous deposits of sulphur of great economic importance. Individual deposits are found in many countries and in the aggregate contain substantial reserves. Some have attained importance commercially, but a considerable number are in isolated regions, and costs for production and transportation therefore are excessive.

*Formations.*—Most of these deposits result from thermal activity associated either with solfataras or thermal springs. The gases and solutions given off, being highly acid, have altered the formations surrounding each vent into masses of bleached, porous rock. The sulphur was deposited here subsequently, both in the crystalline and massive state, filling the fissures, vugs and pores. The bleached outcrops of the altered rock indicate the presence of the ore, which, because of the need of oxygen to effect deposition, is usually found at shallow depths.

*Distribution.*—Scattered deposits lie in the mountain ranges bordering the Pacific Ocean, including the Aleutian Islands of Alaska,<sup>38</sup> the Sierra



Nevada mountains of California,<sup>2</sup> numerous mountain ranges of Mexico,<sup>3</sup> and the Andes of South America.<sup>22,23</sup> Other localities in the United States where deposits have been operated intermittently include the states of Nevada,<sup>1,28</sup> Utah,<sup>35</sup> Wyoming,<sup>74,75</sup> New Mexico, and the western part of Texas.<sup>48</sup> Bordering the Pacific Ocean on the west are the sulphur deposits of Japan,<sup>81</sup> where more than 30 small mines are in operation, and deposits have been recently found and developed in Java. In Europe, Spain<sup>71</sup> produces sulphur from small deposits in limestone, and production is being actively developed by the Soviet Government from deposits scattered throughout European and Asiatic Russia.

*Mining and Preparation.*—Mining methods consist of simple forms of open-pit, gophering, room-and-pillar, and open-stope and pillar. Methods of beneficiation include the liquefaction of sulphur by bringing the ore in contact with steam in pressure retorts or autoclaves, recovery by this method rarely exceeding 50 per cent. Low-grade ores sometimes may be concentrated by flotation before additional treatment. In Japan and Chile distillation of sulphur is commonly practiced by charging the ore into retorts of small capacity with the heat applied externally. Usually six to twelve retorts comprise a unit, equipped with common firebox, flue, and condensation chamber where the distilled vapors are condensed to flowers or lump sulphur.

#### ORIGIN OF SULPHUR

The principal deposits of the world—the salt-dome type and those of Sicily—have several features in common, which may be indicative of their origin. In both regions sulphur occurs in or is closely associated with limestone, gypsum or anhydrite; carbonaceous matter, in the form of petroleum and petroleum residues, or as bitumen, is a frequent constituent of the sulphur-bearing rock or associated formations; and hydrogen sulphide is present as a gas or in solution in water.<sup>62</sup> These characteristic features led to the hypothesis that sulphur in these deposits was formed through the reduction of sulphates by carbonaceous matter and that, as a result of this reaction, calcium carbonate and hydrogen sulphide were first formed, the hydrogen sulphide being subsequently oxidized to sulphur. Laboratory experiments have shown that this reaction requires temperatures in excess of 1100° F., and there are no present indications that such temperature existed in either type of deposits. The question arises as to whether time, measured in geologic sense, may at normal temperatures bring about reactions that in the laboratory require high temperatures. Another theory, recently discussed in various publications, does not require unusual temperatures, but is based on the power of certain species of anaerobic bacteria to reduce mineral sulphates.<sup>7,19</sup> These organisms derive energy from carbonaceous

matter of their environment and oxygen from molecular oxygen of the sulphate minerals. According to this theory the first product of reduction is calcium sulphide, which reacts in the presence of water with carbon dioxide, generated in the life process, to form calcium carbonate and hydrogen sulphide.<sup>29</sup> The oxidation of hydrogen sulphide to sulphur can best be explained by the assumption that this reaction took place where deposits were exposed to oxidizing conditions. This assumption has been made for Sicilian deposits, and there is no evidence available or recorded that would prohibit a similar consideration for the salt-dome type.

Volcanic emanations<sup>11</sup> frequently contain sulphur vapors, sulphur dioxide and hydrogen sulphide. Many deposits that outcrop at the surface have evidently originated from one or more of these gases. Temperatures are prohibitively high at the seat of activities for deposition, but at more remote locations, or during dying stages of volcanism, lower temperatures are more favorable. There are three methods by which sulphur may be deposited from solfataras: the first by condensation of sulphur vapors, the second by reaction between hydrogen sulphide and sulphur dioxide, and the third by oxidation of hydrogen sulphide. Deposits may also originate by deposition of sulphur from thermal springs containing hydrogen sulphide. Other agents of deposition,<sup>29</sup> active in mineral springs, are sulphur bacteria, which have the power of oxidizing hydrogen sulphide to sulphuric acid and, in an excess of the gas, of storing sulphur in their cells.

#### PYRITES

*Definition.*—Pyrites is an inclusive term used to designate metallic sulphides such as iron pyrites (pyrite), magnetic pyrites (pyrrhotite), white iron pyrites (marcasite), copper pyrites (chalcopyrite), and tin pyrites (stannite). The metallurgical treatment of some of these and other sulphide minerals in the copper, lead and zinc industries frequently results in the recovery of by-product sulphur in some form. The term pyrites is here largely limited to sulphide minerals of which the principal value is sulphur. The minerals of common commercial interest under this definition are pyrite, pyrrhotite and marcasite.

*Properties.*—Pyrite ( $\text{FeS}_2$ ) when pure contains 53.4 per cent S and 46.6 per cent Fe. It has a brass-yellow color, metallic luster, greenish or brownish black streak; and crystals of cubic, octahedral, pyritohedral, or other isometric forms are common. The hardness is from 6.0 to 6.5 and the specific gravity 4.9 to 5.2. It has no cleavage, is brittle, and of uneven fracture.<sup>18</sup>

Pyrrhotite, varying in chemical composition, is usually accepted as  $\text{Fe}_{11}\text{S}_{12}$ , and, if pure, contains 38.4 per cent S and 61.6 per cent Fe. It

is bronze in color, has metallic luster and black streak. Usually it occurs in a massive form but occasionally in tabular or pyramidal crystals of the hexagonal system. Its hardness is from 3.5 to 4.0, and it has a specific gravity of 4.5 to 4.6. It is brittle, of uneven fracture, and is magnetic.

Marcasite has the same chemical composition and hardness as pyrite. The luster is metallic, the color pale yellow to almost white. It is opaque, with a greenish black streak. It is sometimes found in tabular crystals of the orthorhombic system, also frequently in radiating fibrous masses, and its multiple twinning often results in jagged outlines. It is brittle, of uneven fracture, and has a specific gravity of 4.85 to 4.90.

*Origin.*—Because pyrite is the most common sulphide mineral, associated with rocks of all ages and types, particularly with sulphides of copper, lead and zinc, a discussion of its origin here must necessarily be limited to deposits where the mineral is found in large masses of relatively pure pyrite or pyrrhotite. The origin of such deposits is generally ascribed to their connection with the intrusion of igneous rocks.<sup>36</sup> Literature on the subject indicates that the origin is dissimilar in other respects and divides these deposits into two broad groups—those formed at high temperatures and those formed at intermediate temperatures. Associated minerals are used as evidence of the temperature at which deposition took place. Those associated with silicates and pyrrhotite were formed apparently at relatively high temperatures and at great depths. In this class are included deposits of magmatic origin, those of the contact-metamorphic type, and those believed by many to have resulted from the injection of sulphide magmas. The second group, those formed at intermediate temperatures, includes deposits associated with minerals such as calcite, barite and quartz. Deposits of this type may have been formed by hydrothermal replacement at moderate depths, and the origin of deposits in Spain has thus been explained.<sup>8,70</sup>

#### PYRITES DEPOSITS

##### UNITED STATES

Deposits are being operated in Tennessee, New York, Missouri, Virginia, California, Wisconsin, Montana and Colorado. The principal production is from the Ducktown region, in the most southeasterly corner of Tennessee.

Pyrites, under the name of "coal brasses," is sometimes recovered in the preparation of certain coals, for market, especially those of the interior field.

*Formations.*—The Ducktown district lies in the mountainous area of the southern Appalachians. The formations are of Lower Cambrian age, and consist almost wholly of metamorphosed conglomerate sand-

stone and shale, complexly folded and faulted. For many years the ore bodies were considered to be the result of partial replacement of included sedimentary beds of limestone and marble,<sup>15</sup> but recent investigation resulted in the theory that the deposits were formed by a series of mineral depositions, each initiated by fault movements, and each characterized by a distinct group of minerals. The minerals of each succeeding stage replaced those of the preceding stages, so that the ore minerals, which were introduced last, replaced most of the vein material and portions of the country rock.<sup>53</sup> The ore bodies are elongated, roughly tabular masses, some of which are curved, folded or lens shape. In general the veins strike northeast and dip from 35° to 65°. In length they extend over a distance of about 2000 ft. with widths from a few feet to a maximum of 180 ft., and they have been worked to depths of 1600 ft.<sup>40</sup> The ore consists principally of massive pyrrhotite, pyrite and chalcopyrite, with minor amounts of sphalerite and galena. The chief gangue minerals are quartz, actinolite and tremolite. The ore contains from 25 to 39 per cent S, 35 to 45 per cent Fe, and 0.8 to 2.5 per cent Cu.

*Mining.*—The ore is mined entirely by underground methods. Sub-level stoping is employed in the mines of the Tennessee Copper Co.<sup>40</sup> Where the ore is not wider than 40 ft., longitudinal sublevel stopes are taken out from wall to wall. In wider sections transverse stopes 40 ft. wide alternate with pillars of equal thickness. The ore, when broken in stopes, falls to funneled raises and is then worked through grizzlies into cars on the haulage level. After tramming to ore pockets or direct to skips it is hoisted to the surface through an inclined shaft in the foot-wall. The Ducktown Chemical and Iron Co., now merged with the Tennessee, employs the room-and-pillar system in wide ore bodies.<sup>32</sup> Rooms 60 ft. square are blocked out with intervening 40-ft. pillars. Development is through raises extending from the haulage level to the floor pillar above. Underhand stoping commences at the top of the raise and is continued by a series of benches around the raise.

*Preparation.*—Commercial interest in Ducktown ores was formerly in the copper, but on account of the strategic location in respect to large acid-consuming centers the sulphur content has become of increasing importance. The ore is treated in plants operated by producers for the recovery of sulphur as sulphuric acid, as well as for the recovery of copper concentrates and iron sinter. Some of the crude ore is smelted directly for its copper content, sulphur gases being sent to the acid plant. The major portion of the ore, usually low in copper, is treated by flotation, whereby copper and pyrites concentrates are separated and recovered. The latter are roasted in Herreshoff furnaces and sulphur dioxide is sent to the chamber or contact acid plants. Calcines from the roaster are sintered and shipped to Birmingham for manufacture of pig iron and steel.

## SPAIN

*Formations.*—The zone of pyrites deposition that extends through the Province of Huelva in southern Spain and into Portugal has furnished the major portion of the world's supply of pyrites for many years (refs. 7 and 70, ref. 30, pp. 635–648). Formations in the vicinity of the deposits consist of folded slates and graywacke, striking east and west, and probably of Carboniferous age. After being folded, the slates, which dip steeply northward, were intruded by porphyries and diabase. The ore bodies are found in the form of elongated lenses in the slate and porphyries or at the contact of intrusive rocks. There are approximately 50 such lenses, the maximum dimensions of which are: length, 6000 ft.; width, 800 ft.; known depth, 1800 ft.<sup>50</sup> The ore bodies usually are defined by a sharp contact with the enclosing rocks, and in these places the ore consists essentially of massive pyrite. Frequently the massive ore is enclosed by a disseminated type in zones of transition. There are also minor amounts of chalcopyrite, sphalerite and galena in the ore. The small amount of gangue rock consists chiefly of quartz and occasionally of barite. It has been estimated that total reserves of Spain approximate 500,000,000 tons. The ore body in the San Dionisio lode, operated by Rio Tinto Company, Ltd., is reported as the largest deposit in the world, having approximate dimensions of: width, 600 ft.; length, 3300 ft. and known depth of 1200 feet.

*Mining.*—The original open cut on San Dionisio lode has resulted in a terraced crater of which the rim is approximately 1350 by 1800 ft. with a depth of 450 ft. Some mining is still being carried on by glory-hole methods in the open pit. The ore is scraped into winzes, which feed loading pockets on the 23d level. From there it is trammed by compressed-air haulage, hoisted electrically in skips to the 16th level, and hauled by electric locomotives through a 3-mile adit to the surface. Former underground methods consisted of driving wide drifts and cross-cuts on approximately 40-ft. centers with 45-ft. vertical intervals between the levels. The present underground system, accounting for the major production, consists of mining the entire block of ore between the old filled stopes by driving sublevels 9 ft. apart across the ore body. From these sublevels the ore is broken out in blocks 9 by 9 by 9 ft. Timber and lagging are used in all workings, the timbers being subsequently and completely removed and the stopes filled with waste. Good ventilation and removal of all timber are necessary on account of fire hazards. Ore from underground workings is dropped through passes to the 16th level, where it is hauled through the adit to the surface.

*Preparation.*—Ore averaging 48 per cent S and less than 1.5 per cent Cu is marketed with no treatment other than screening. Ore containing more than 1.5 per cent Cu is leached for its copper content before ship-

ment. This is accomplished in huge piles, which are intermittently sprayed with water for two or more years. The copper solution is collected and passed through launders containing scrap iron, upon which copper is precipitated. Washed ore analyzes approximately 49 per cent S and 0.4 per cent Cu. When copper content reaches 3.0 per cent, the ore is smelted for recovery of copper, sulphur being lost to the atmosphere. Ore of this grade is also selected for the production of sulphur by a process similar to that used in Norway.

### NORWAY

In Norway, which ranks third in world production, cuprous pyrites production comprises the major mining industry and is the principal source of copper (ref. 30, pp. 605-614). Important deposits are found in the Hardanger, Trondheim, Grong, and northern districts. The Lokken mine, operated by the Orkla Mining Co. with a smelter at Thamshavn, is the major producer of pyrites.

*Formations.*—The Lokken deposits are in the Scandinavian mountain chain and the country rock is described<sup>17</sup> as schistose greenstone with gabbro intrusions on the hanging wall a short distance from ore. The main ore body has the shape of a lens lying in a 45° position with a thickness of 150 ft. in the center portion, and with a length of approximately 6000 ft. A medium grained pyrite containing some quartz is the principal constituent, with sulphur content averaging about 42 per cent and copper 2.5 per cent.

*Mining and Preparation.*—Underground mining is used, in which stoping is accomplished by the shrinkage system. After crushing and screening, the major part of the production is shipped to Scandinavian pulp mills or European acid plants. Ownership of the copper-bearing calcines is retained by the producer, the calcines being reshipped either to a company-owned leaching plant or to Germany for copper recovery and subsequent iron production. Sulphur as such is produced from part of the ore by the Orkla process essentially as follows: A charge consisting of ore, coke, quartz and limestone is smelted in a blast furnace, and the resulting gases contain sulphur vapor, sulphur dioxide, carbon disulphide and carbon oxysulphide.<sup>65</sup> These react in the presence of a catalyst to form sulphur vapor, which is condensed to a liquid and allowed to solidify. A matte containing copper is drawn from the furnace and subsequently converted to blister copper.

### FINLAND

An increase in pyrites production in Finland within the last few years has been due chiefly to the unique operations of the state-owned

Outokumpu mine, which also constitutes an important source of copper. This deposit is situated approximately 30 miles west-northwest of Joensuu.

*Formations.*—Cuprous pyrites are associated with quartzites and schists. Diamond drilling and underground development indicate that the deposit is approximately 7800 ft. long, 6 to 60 ft. wide and about 1200 ft. deep along the dip, which is  $30^{\circ}$  to the south. The valuable constituents of the ore are 4 per cent Cu, 26 per cent S, 26 per cent Fe, and a small amount of gold and silver.

*Mining and Preparation.*—Sublevel stoping methods are used in the narrower portions of the lode and shrinkage stopes in the wider section. After being crushed and ground, the ore is concentrated in a flotation plant. Concentrates, containing approximately 22 per cent Cu, carry the gold and silver values. The pyrites concentrate contains 44 per cent S and 0.4 per cent Cu. Part of the copper concentrate is shipped by rail to sulphuric acid plants near Imatra, where sulphur is recovered as sulphuric acid and calcine is returned to Imatra for smelting with the remainder of the raw copper concentrates. Smelting is accomplished in a circular electric reverberatory furnace, utilizing the high resistivity of the molten slag for the generation of heat. Matte is subsequently converted into blister copper carrying gold and silver values. Sulphur dioxide is removed from smelter and converter gases by washing, cooling and passing through absorption towers where sulphur dioxide is dissolved in basic aluminum sulphate. The cold saturated solution is heated to expel sulphur dioxide. The concentrated sulphur dioxide gas is dried and compressed into a liquid for shipment to pulp mills in Finland. The pyrites concentrate is shipped to other pulp mills for recovery of sulphur dioxide and calcines are purchased by a steel company.

#### OTHER DEPOSITS

In Japan, which is the second largest producer of pyrites, the entire output is consumed locally in the manufacture of acid. Italy, which ranks fourth in the production of pyrites, is similar to Japan in that its production is principally for local consumption, and the recovery of iron from the calcines is an important factor because of the scarcity of iron ores. Other important sources of pyrites are Russia, Germany, Greece, France, Cyprus, Sweden and Canada. Countries in which minor amounts of pyrites are being produced include Chosen, Yugoslavia, Czechoslovakia, Union of South Africa, Algeria, Australia, Poland, South Rhodesia, Rumania and the United Kingdom.

#### MARKETING AND USES

The largest single use of sulphur is for the production of sulphuric acid, and native sulphur and pyrites are both used for this purpose.

The manufacturer of sulphuric acid, therefore, has his choice between these two minerals, and his selection will be determined by economic factors such as price, availability and dependability.<sup>16</sup> Sulphur and sulphuric acid enter into a vast number of industries and a multiplicity of uses, some of which are shown in Fig. 4.

In the preparation of fertilizers washed phosphate rock is treated with sulphuric acid and this not only makes the phosphate available to plants but furnishes sulphur, which is also an essential plant food. In petroleum refining sulphuric acid removes gums and tarry matter as well as other

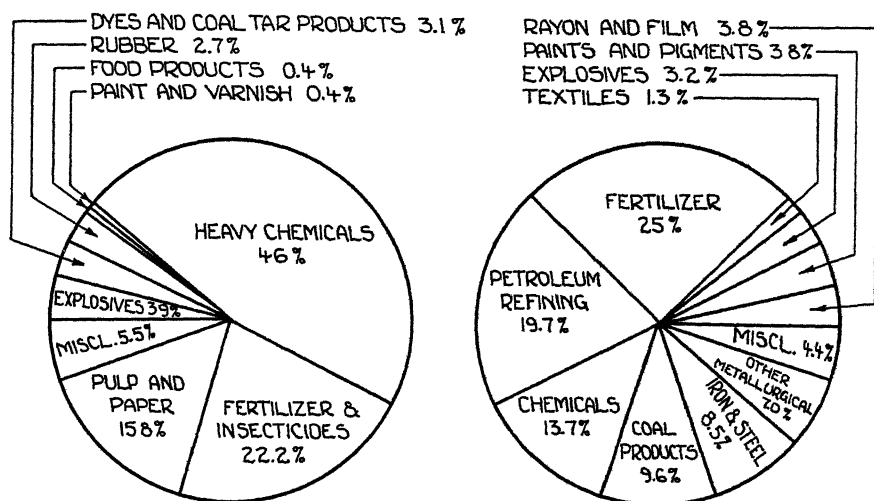


FIG. 4.—DISTRIBUTION BY INDUSTRIES OF (a) SULPHUR AND (b) SULPHURIC ACID USED IN THE UNITED STATES IN 1934.

From figures assembled by *Chemical and Metallurgical Engineering*.

constituents that would form end products of a corrosive nature. Nitrogenous constituents resulting from the coking of coal pass off as ammonia and the gases are scrubbed with sulphuric acid to produce ammonium sulphate. Sulphuric acid is used in the purification of other products of coal distillation, such as benzol and toluol. The iron and steel industry uses sulphuric acid mainly for pickling of iron and steel products before galvanizing. Another metallurgical operation consuming sulphuric acid is electrolytic production of zinc. A large and increasing use in the paint and pigment industry is for manufacture of titanium dioxide. The ore is dissolved in sulphuric acid and titanium dioxide is then precipitated in a pure form by hydrolysis. Black powder, including sulphur as a constituent, has been almost entirely replaced by nitroglycerin in the form of dynamite and similar explosives. In the manufacture of nitroglycerin nitration is carried out with nitric acid in the presence of concentrated sulphuric acid. Explosives for military use contain large amounts of



nitrocotton, which is made by nitrating cellulose in a bath of mixed nitric and sulphuric acids. In the viscose process for making rayon the viscose solution is precipitated in a sulphuric acid solution. Sulphur is employed in the viscose process in the form of carbon bisulphide, this being used to make cellulose xanthate from caustic soda and cellulose. Large amounts of sulphuric acid are used in the nitrating bath in making nitrocotton from which motion picture films are produced. The textile industry uses sulphuric acid in the various operations of washing, bleaching, shrinking and dyeing.

As in the manufacture of sulphuric acid, either sulphur or pyrites may be used as a raw material for the production of sulphite pulp. In this process sulphur dioxide is absorbed by milk of lime solution or in towers filled with limerock, and the resulting solution containing calcium bisulphite and sulphurous acid digests the wood chips until most of the noncellulosic materials have been dissolved. In some Kraft plants sulphur and caustic soda are used instead of sodium sulphate in the belief that this modification is productive of a stronger fiber.

The amount of sulphur used for the manufacture of rubber depends upon degree of hardness desired in the final product. Hard rubber in storage-battery cases usually contains 30 per cent, while rubber used in tire stock may contain not more than 1.5 per cent. Sulphur is also consumed in the manufacture of newer synthetic rubbers, which are claimed to have more resistance to chemical attack and oil deterioration than natural rubber. Thiokol, one of the most interesting of new rubbers, results from the chemical reaction between ethylene dichloride and a solution of sodium polysulphide.

Sulphur and sulphuric acid are assets of great and expanding value in agriculture. The insecticidal and fungicidal uses of sulphur are being rapidly expanded by the work of experiment stations and research organizations. Sulphur, in the form of dust and also in the form of spray materials, such as lime sulphur solution and wettable sulphurs, is used in control of pests that attack fruit trees as well as field and truck crops. Of recent interest is the development of combination contact insecticides, such as mixtures of pyrethrum and rotenone with sulphur. Sulphur and sulphuric acid are used as soil amendments to neutralize alkalinity and to correct sulphur deficiency. Where soils contain a relatively high concentration of selenium, together with a deficiency in sulphur, the selenium may be selectively absorbed by certain forms of plant life and cattle forage thus poisoned. Recent experimental work tends to show that addition of sulphur to such soils will prevent absorption of selenium by plant life. Sulphur in the form of sulphur dioxide is used in greenhouses and mushroom houses. Carbon bisulphide serves as a fumigant for stored grains and is also used to exterminate insect pests in the soil and as a weed killer. Sulphuric acid of about 10 per cent concentration

is gaining steadily as a weed killer, particularly for cereal crops. Concentrated sulphuric acid is being used for delinting cottonseed before planting. Sulphur incorporated in feed is under investigation as an intestinal disinfectant for poultry, and at present it offers great promise as a protection against parasitic organisms that annually cause enormous losses to the industry.

#### TESTS AND SPECIFICATIONS

Specifications for sulphur and pyrites concern three items of principal interest: (1) that the raw material shall contain a guaranteed minimum of sulphur; (2) that it shall contain not more than specified amounts of recognizedly injurious materials; and (3) that there shall be present not more than certain amounts of materials that prevent or retard combustion.

Sulphur produced by the Frasch process frequently averages 99.8 per cent, is guaranteed to contain not less than 99.5 per cent S, and to be free of arsenic and selenium. Impurities consist of traces of ash and petroleum hydrocarbons. Italian sulphur is sold under several different grades as a result of the varying amounts of inorganic impurities.

Commercial flour sulphur is prepared by grinding and sizing crude sulphur, and the chief specification for this material, in addition to those listed above, relates to particle size. Commercial flour sulphur used by the rubber industry, in addition to being carefully sized, should contain not more than 0.01 per cent free acid. Flowers of sulphur is sublimed crude and should contain not less than 30 per cent amorphous (insoluble in carbon bisulphide) sulphur. The purpose of this specification is to ensure that the product has been sublimed and not because amorphous content is advantageous.

To ensure the proper specifications sulphurs, as marketed, are customarily tested for moisture, ash, sulphur, organic impurities and free acid. Moisture and ash are determined by standard methods. Dry sulphur is extracted by carbon bisulphide and the sulphur content determined by subtracting the insoluble portion from 100. When amorphous sulphur is present hot aniline is used as an additional solvent. Organic material is generally determined by burning a small sample under controlled conditions and comparing the resulting deposit of carbon with a series of standards. Free acid is determined by wetting a ground sample with alcohol and extracting the acid with water, subsequently titrating the extract with  $\frac{1}{40}$  normal caustic solution.<sup>57</sup>

Because injurious impurities are more common in pyrites than in sulphur, specifications are more rigid. Specifications for pyrites are usually determined by agreement under long-term contracts between the producer and consumer, because each mine produces ore peculiar to

itself. Physically pyrites is classed as one of two grades, lump or fines. Lump ore varies in size from 2 to 10 in. and should contain not more than 10 per cent through  $\frac{1}{2}$  or  $\frac{3}{8}$ -in. screen. The undersize, classed as fines, was sold at a discount prior to development of suitable burners. Contracts usually call for a premium or penalty when the sulphur content deviates from a guaranteed percentage. Zinc and lead are objectionable because they cause retention of sulphur in calcines. In the manufacture of sulphuric acid more than 1 per cent of arsenic is objectionable, and the rigidity of this specification depends on the type of equipment available at consumers' plants for arsenic removal from the gases. Where pyrites is to be used for papermaking, selenium may cause oxidation of the sulphur dioxide to sulphur trioxide in the digester, and is therefore objectionable. The disposal of calcines, which may contain other valuable constituents, is by agreement between producer and consumer.<sup>60</sup>

Test methods employed are the usual standard methods for determination of sulphur, iron, copper, zinc, lead, arsenic, selenium and moisture.<sup>67</sup>

#### POLITICAL AND COMMERCIAL CONTROL

In order to clarify the present status of the world sulphur industry, it is advisable to review major developments during the past 35 years. In the early part of the twentieth century the Frasch process made rapid strides commercially, supplanting Italian and Japanese sulphur in American markets and exporting small amounts. Subsequent to the Armistice, with a greatly increased production and with previously scarce shipping facilities then available, American sulphur gained rapidly in all world markets. During the World War domestic manufacturers of sulphuric acid had difficulty in obtaining pyrites from foreign sources and many plants were altered to burn sulphur. There has been no subsequent change in the attitude of American consumers, because domestic sulphur offers greater security in supply and availability. After the World War American producers, in order to develop additional outlets, found it necessary to enter foreign markets more aggressively, and in 1922 the Sulphur Export Corporation was formed to distribute sulphur in world markets.

Many nations of the world have fostered local sources of production by bounties, tariffs, embargoes and sometimes by complete government control. The Italian Government authorized a subsidy of 20,000,000 lire to cover a period of 18 months, ending June 1935, and by a subsequent decree this subsidy was extended for a two-year period, beginning July 1935, the sum of 30,000,000 lire being appropriated. The purpose of these subsidies is to meet any difference between a minimum price, guaranteed to producers by the Italian Government, and the sales price realized in world markets. Japan protects its industry by a 20 per cent

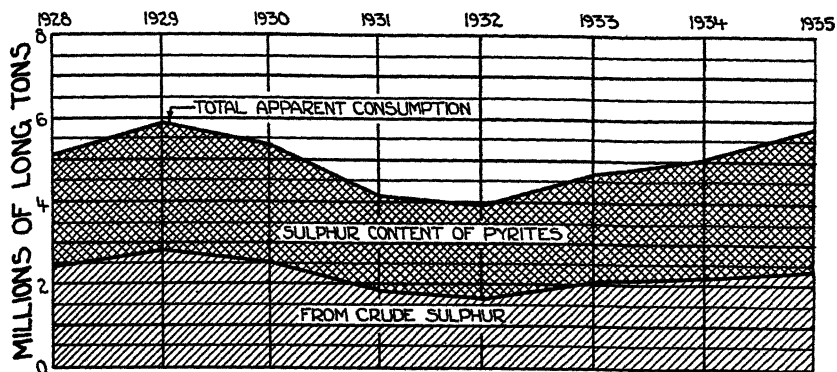


FIG. 5.—WORLD CONSUMPTION OF SULPHUR FROM CRUDE SULPHUR AND PYRITES.

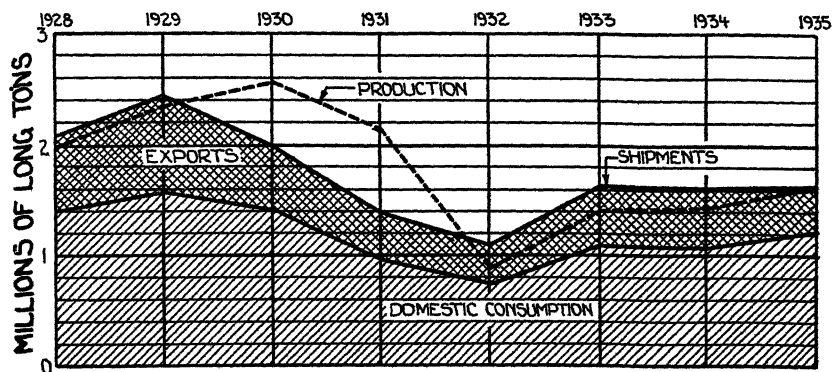


FIG. 6.—PRODUCTION AND SHIPMENTS CRUDE SULPHUR, UNITED STATES.

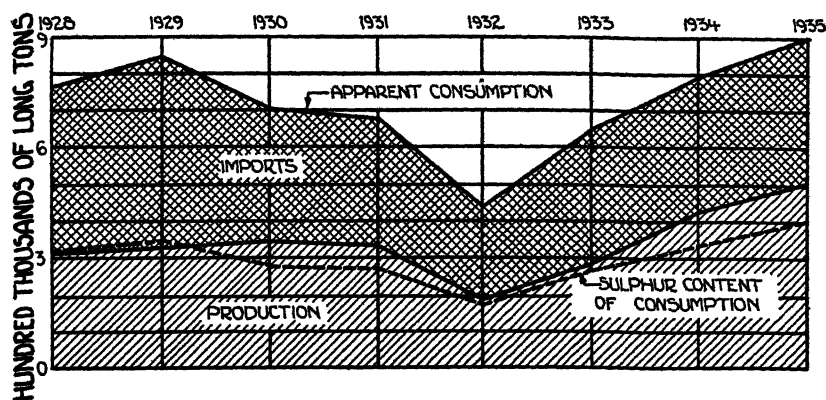


FIG. 7.—DOMESTIC CONSUMPTION PYRITES, UNITED STATES.

duty on the c.i.f. price of imported sulphur. In 1934 the Spanish Government passed a decree prohibiting all imports of sulphur until the price reached the equivalent of approximately \$40 per ton, and this

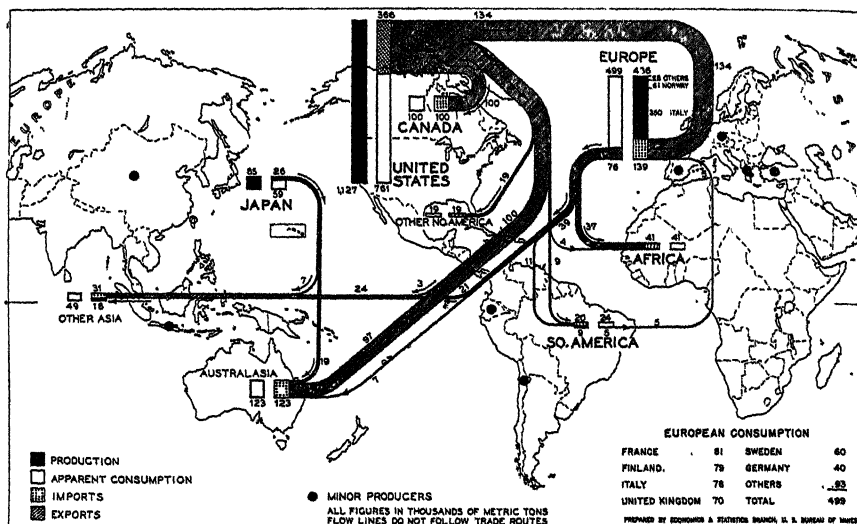


FIG. 8.—WORLD PRODUCTION, CONSUMPTION AND PRINCIPAL TRADE RELATIONS OF SULPHUR IN 1932.

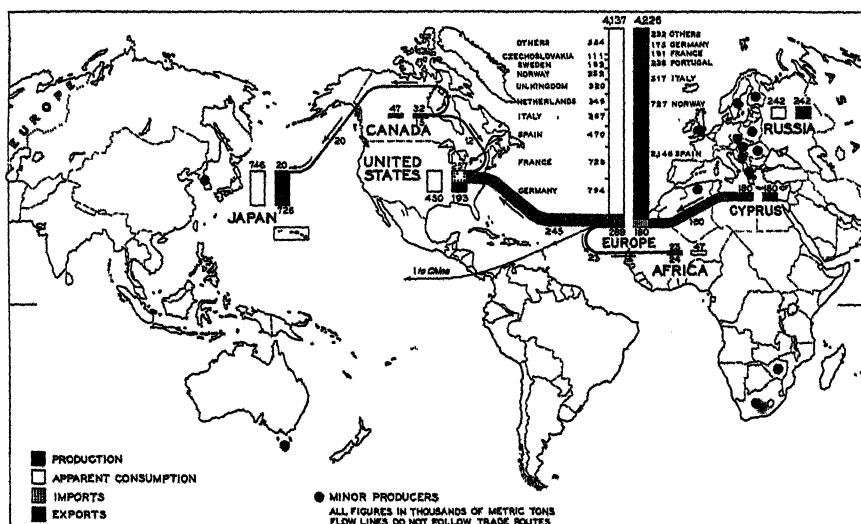


FIG. 9.—WORLD PRODUCTION, CONSUMPTION AND PRINCIPAL TRADE RELATIONS OF PYRITES IN 1932.

serves to protect the new plant in which sulphur is produced from pyrites. Through a government agency, known as the Mining Credit Institute, Chile is authorized to pay a subsidy of 100 pesos per ton of

refined sulphur exported. In Finland a pyrites mine owned and operated by the Government and with a productive capacity equivalent to 35,000 tons of sulphur per year sells pyrites concentrates to Scandinavian paper mills. In Australia there is no sulphur production and the Government pays a bounty of 36 shillings per ton of sulphur equivalent produced from sulphide ores. In Portugal a plant has been built to produce sulphur from pyrites with a capacity about equal to domestic consumption. The industry is protected by an import duty of 0.80 gold escudos per ton plus an import surtax of 20 per cent. The Turkish Government in 1934 placed a heavy import duty on sulphur and at the same time proclaimed that only the domestic product could be used. Other countries have import duties on crude sulphur, not to protect the domestic industry but as a source of revenue.

### CONSUMPTION AND PRODUCTION

Statistics and trends of the sulphur industry are illustrated by Figs. 5 to 7. The part played by sulphur and pyrites respectively in supplying the world requirements for sulphur in all forms is shown in Fig. 5. The adjustment of domestic production to shipments since 1932 is illustrated in Fig. 6, which also shows the failure of exports to increase comparably with domestic consumption in recent years. Increasing domestic production of pyrites is shown by Fig. 7. Charts of world trade relations for the two minerals (Figs. 8 and 9) emphasize the fact that, under present economic conditions, the United States relies on crude sulphur and Europe on pyrites as the principal source of sulphur.<sup>42,52</sup>

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## CHAPTER XLVI

### TALC, SOAPSTONE, AND PYROPHYLLITE

BY JOSEPH L. GILLSON,\* MEMBER A.I.M.E.

**T**ALC belongs to that small but interesting group of minerals having such a low degree of hardness that they feel greasy, and at the same time exhibit an eminent pinacoidal cleavage. According to Foshag and Wherry,<sup>18</sup> the chemical constitution of talc has been studied by a number of investigators without definite conclusions. The analyses show that the ratio Mg:Si varies from 1:1 to 4:3 and the water content from 3 to over 7 per cent. Most reference books on mineralogy give the formula as  $\text{H}_2\text{Mg}_3(\text{SiO}_3)_4$ . According to this, the mineral should carry 63.5 per cent  $\text{SiO}_2$ , but published analyses show a range from 56.86 to 62.10.  $\text{MgO}$  should form 31.8 per cent in the 1:3:4 type, but actual analyses vary from 27.9 to 32.40. Alumina, ferric and ferrous iron, manganese and lime are reported in various analyses, although the purity of the material analyzed may be open to question in some cases.

Part of the water present in some varieties can be eliminated without a notable change in the optical properties of the mineral. This loosely held water is supposed to be held along the basal cleavage planes by electrostatic attraction. Another talc containing only firmly bound water and approximating the composition 1:4:5 was investigated by Clarke and Schneider,<sup>16</sup> and when the water was driven off, the mineral was decomposed.

The crystal structure of talc has been studied by Gruner.<sup>19</sup> Talc and pyrophyllite have the space group  $\text{C}_{\frac{6}{2h}}$ . The unit cell contains four molecules of  $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$  or  $\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2$ , respectively. For talc,  $a$ ,  $b$  and  $c$  in Ångstrom units are 5.26, 9.10 and 18.81. Beta is  $80^\circ$ . For pyrophyllite:  $a$ ,  $b$  and  $c$  are 5.14, 8.90 and 18.55, with an angle for beta of  $80^\circ 5'$ .

The similarity of crystal structure of talc and pyrophyllite explains the close similarity of the minerals in physical and optical properties. The two minerals are distinguished only with difficulty under the polarizing microscope. The optic angle of pyrophyllite is larger than that of talc, and the indices of refraction are about 0.005 higher. Pyrophyllite is a

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<sup>18</sup> References are at the end of the chapter.

trifle harder than talc. The name agalmatolite was assigned by the mineralogist Klaproth to the Chinese figures carved from a compact pyrophyllite, the name having been taken from the Greek word "agalma," meaning an image. The name has persisted in some districts.

Studies of the thermal decomposition of talc were made by Kôzu and Masuda.<sup>20</sup> The water is given off between 900° and 1050° C. The conversion of talc to enstatite and clinoenstatite and cristobalite on heating was investigated by Ewell<sup>17</sup> and his associates at the National Bureau of Standards. Water was driven off in excess of one molecule between 380° and 500° C., but this caused no change in crystal structure. The molecule of combined water was driven off between 800° and 840° C. This water loss was accompanied by a large endothermic heat effect and an increase in specific gravity from 2.83 to 2.91, and by breakdown of the talc to enstatite and amorphous silica. Inversion of the enstatite to clinoenstatite took place gradually, but was complete in material heated to 1300° C. At this temperature the silica was converted to cristobalite. The specific gravity of the completely converted product is 3.01.

According to new data on dielectric constants of mineral powders assembled by Rosenholtz and Smith,<sup>22</sup> talc has a constant of 9.41.

The characteristics of soapstone and steatite are so varied that no simple definition can be applied to describe these rocks. The name "steatite" seems in general to be used to describe a massive stone comparatively rich in talc, whereas soapstone is a name applied to almost any massive soft rock that is readily cut, although it may contain only a small percentage of the mineral talc. Usually a soapstone is an impure talc rock that is sufficiently massive to be quarried in large pieces, but is too low in grade to produce a marketable talc. The other minerals present in most examples are dolomite or magnesite, chlorite, antigorite and magnetite.

#### DISTRIBUTION AND MODE OF OCCURRENCE

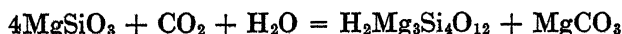
Commercial deposits of talc and soapstone are found only in much altered, old ultrabasic intrusions, or in certain metamorphosed dolomitic limestones. Thus, talc and soapstone deposits are confined to the pre-Cambrian rocks, or to the much metamorphosed members of younger series. In metamorphic areas, talc and soapstone are by no means uncommon; hence, nearly every large area underlain by metamorphic rocks contains some talc. Since many countries of the world include such areas, the list of countries with talc and soapstone deposits is very long. Deposits occur up and down the piedmont of both Americas, in the folded and metamorphosed masses of the Alps and the Pyrenees, in Piedmont of Italy, in Russia, Scandinavia, South Africa, India, Manchuria, China, Japan, and the folded west coast of the Americas.

Pyrophyllite deposits are not nearly so numerous as those of talc. It is possible that some of the less well-known talc deposits may actually contain pyrophyllite instead of talc. A deposit at Tschistaya-gora<sup>85</sup> in the Southern Urals had been worked as a talc deposit, but when a mineralogical investigation was made, the material proved to be pyrophyllite and no talc is present. Large pyrophyllite deposits occur in North Carolina,<sup>85</sup> and an occurrence in clay has been described recently from San Diego County, California.<sup>89</sup>

Deposits are reported to occur in China and Japan (Korea). In Korea,<sup>10</sup> the pyrophyllite occurs in granophyre (i.e., rhyolite) and tuffs. It is produced at Kasato in Chinto-gun and at Ogmesean and Ibam in Kainan-gun.<sup>77</sup> The deposits in China<sup>51</sup> have not been described in detail in publications available to the writer.

Pyrophyllite occurs, but not in workable quantities, in Hungary, Saxony, Scotland, Turkestan, Newfoundland and British Columbia.

Most of the talc occurrences of the world have so many common characteristics that it is evident that talc is the result of a certain type of alteration of rocks carrying magnesia, or of magnesia minerals formed in a low-magnesia rock by solutions that preceded the solutions that formed the talc. It occurs in altered basic igneous rocks, or in contact zones close to basic igneous rocks, or in metamorphic limestones and dolomites in which actinolite, tremolite or enstatite had previously been formed. Lindgren\* states that talc may be formed from any magnesian amphibole or pyroxene by the action of H<sub>2</sub>O and CO<sub>2</sub> according to the formula:



In reports where the paragenesis of mineral formation has been given, talc is a late mineral in the sequence, having formed at moderate temperature from hydrothermal solutions given off from an intrusion in depth. It is never a weathering mineral, nor does it ever seem to have formed from the simple serpentinization of basic intrusives. Where present in serpentine rocks, actinolite and biotite had formed in the serpentine prior to the formation of the talc. The talc is pseudomorphic after the actinolite, or after chlorite which replaced the biotite.

Harker† considers that "talc represents a more advanced metamorphism in the purely dynamical sense; or in other words, talc is more emphatically a stress mineral than the serpentines." Although the writer does not imply that talc could not form from simple dynamic metamorphism of a basic igneous rock, he has not, in his own observations, ever seen any talc deposits that appear to have formed in that way.

\* W. Lindgren: *Mineral Deposits*, 391. New York, 1933. McGraw-Hill Book Co.

† A. Harker: *Metamorphism*, 275. 1932. Methuen and Co. Ltd.

Hess<sup>7</sup> concludes that shearing has simply facilitated the later access of solutions.

Lindgren (*op. cit.*) states that talc is very rarely found in ordinary ore deposits. It does occur in some ore deposits, but its association in them seems to present no dissimilar features from the normal talc deposit. Interesting examples of talc as a gangue mineral in ore deposits are The Gaika Gold Mine,<sup>24</sup> at Que Que in Southern Rhodesia; the Beatrice Mine at Selibin in the Federated Malay States,<sup>25</sup> in the pyrrhotite chalcopyrite ores at Ducktown, Tenn.,<sup>26</sup> and the zinc ores in St. Lawrence County, New York.<sup>27</sup>

Talc occurs in minor quantities in many places, but its association in these is similar to that in the commercial talc deposits. Larsen<sup>11</sup> has described the occurrences of talc in the corundum deposits of North Carolina and in the serpentine at Brinton's Quarry, West Chester, Pa. In these deposits in altered basic igneous rocks, distinct bands occur of talc, anthophyllite or enstatite, vermiculite or chlorite, depending upon the locality. The talc is related to the action of the solutions that formed albitites, which are found in the serpentines.

Hess,<sup>7</sup> who has made a thorough study of the serpentines of the North American piedmont, concludes:

The evidence is absolutely conclusive that steatitization is always younger than serpentinization and unrelated to it in any way. Minerals formed during steatitization may always be found replacing serpentine, but serpentine never replaces any of these minerals.

The recognition of the talc in dolomite as a contact-metamorphic phenomenon dates back nearly 50 years, when Weinschenk<sup>58</sup> described the deposits near Wunsiedel in Bavaria.

According to Stuckey,<sup>91</sup> the North Carolina pyrophyllite deposits are metasomatic replacements of acid tuffs and breccias of both dacitic and rhyolitic composition. The solutions presumably came from a large intrusive in depth. Clapp and Buddington studied volcanic rocks altered to pyrophyllite, and both concluded the mineral had formed by hot juvenile waters under conditions of moderate temperature and pressure.

It is not possible in a paper of this length to describe the geological features of every important talc deposit. A few will be selected as representative.

#### TALC DEPOSITS

*In Carbonate Rocks.*—Some of the best quality talc produced in the world comes from the deposits in metamorphosed dolomites intruded by igneous rocks. Examples of such deposits are those at Madoc in Ontario; St. Lawrence County, New York; those deposits in North Carolina and Georgia associated with the Murphy marble of Cambrian age; deposits in California; Pennsylvania and New Jersey; in the Fichtelgebirge of Bavaria; in Uruguay, Austria; Rumania; and India.

The Madoc district<sup>44</sup> in Ontario lies in Hastings County, on the north shore of Lake Ontario, and close to the border of the Laurentian Highlands. One belt of six separate talc deposits, lying east of Madoc village, is about one mile long. Another, of no commercial importance, is found near Eldorado, 6 miles to the north. The rock in which the talc occurs is dolomite belonging to the Grenville-Hastings series, and the deposits are found within  $\frac{1}{2}$  mile of the visible contact with the Moira granite batholith. The dolomite contains tremolite and brown tourmaline, which had formed as a result of the contact metamorphism resulting from the granitic intrusion.

The talc deposits in St. Lawrence County, New York,<sup>34</sup> are similar to those in the Madoc district of Ontario. The same Grenville limestone was contact-metamorphosed by an intrusion of a porphyritic granite exposed near by, and the sediment contains typical contact-metamorphic minerals, phlogopite, graphite, diopside and scapolite, enstatite, and a great deal of tremolite. The talc occurs in a zone in which the tremolite was very abundant. Further alteration had changed the tremolite to talc. The talc bodies are lenticular masses in series along the strike. They dip uniformly to the northwest at angles of  $30^{\circ}$  to  $60^{\circ}$ . Some 20 separate deposits are known in this belt, which lies in the towns of Fowler and Edwards. The masses of talc are very large. In the Arnold mine of the W. H. Loomis Talc Co., the talc forms 124 ft. out of 146 ft. measured from footwall to hanging wall.

An excellent grade of talc has been produced since 1859 from the Murphy marble in the extreme southwest corner of North Carolina<sup>35</sup> and that part of Georgia,<sup>32</sup> contiguous along the strike. Producing centers are Nantahala, Marble, Regal, Culberson and Murphy, North Carolina, and Chatsworth, Georgia. The talc occurs as lenses in the marble and, as at Murphy, lies against a quartzite footwall or hanging wall near the contact; but in the marble, tremolite in crystals up to  $\frac{1}{4}$  in. in diameter have been found. Near the surface the rock and talc are badly weathered, and masses of talc are recovered in clay.

*In and Adjacent to Serpentinized Ultra Basics.*—Talc deposits and soapstones associated with serpentines are more numerous than those in carbonate rocks. Speaking broadly, the individual deposits are smaller, and the quality of the talc is not so high as the former type. Outstanding exceptions occur both as to size and quality. The deposits in Vermont, some in North Carolina, Georgia and Washington, in France, Italy, Spain, South Africa, etc., belong to this type. The soapstone deposits of Virginia differ in that the ultrabasic was never serpentinized. Although talc is commonly associated with serpentines, it is now rather generally recognized that the serpentinization of the basic intrusion was one process, which in some cases was automorphic, and which in others may have resulted from solutions accompanying regional deformation; and

the steatitization was another and later process. The Vermont talc deposits illustrate this type.<sup>37</sup> They occur in serpentines, or in schists or gneisses at the contacts. The writer, in an earlier publication, summarized the character of these deposits.<sup>37</sup>

One of the largest operations for soapstone and low-grade ground talc in the United States is conducted at Schuyler, Va.,<sup>38-41</sup> by the Virginia Alberene Corporation. The soapstone occurs in altered intrusions of a basic igneous rock in the Loudon schist. The bodies are about 180 ft. thick, and dip to the east about 45°. They are sheetlike in form, and have a trend of about N.30°E. The alteration of the basic intrusions was accomplished by solutions from an intrusive acid rock, and during the period of progressively lowering temperature, the original picrite (a rock consisting of olivine and augite) was altered in turn to green hornblende, actinolite, chlorite and then talc. Hess<sup>38</sup> states that the presence in the original rock of sufficient alumina to permit a good development of the chlorite greenstone facies has resulted in the formation of a soapstone that has enough flexible shreds of relic chlorite to make it durable but still easy to cut.

*In Crystalline Schists.*—A type of talc deposit described by European geologists, and said to occur at many places on the Continent, occurs interlaminated with mica schists. The descriptions of such deposits in the literature are very brief, and the writer questions whether these deposits are not actually replacements of limestone or dolomite lenses in the schists, or of serpentines. For example, the largest deposit in Europe, near Luzenac, in the Department of Ariège on the north slope of the Pyrenees, is described as two beds interbedded with mica schists and Silurian schists. The beds have a length of several kilometers and a thickness varying between 15 and 60 m. The talc contains calcareous inclusions (original dolomite) and also granite blocks. A number of other deposits are listed by Dammer and Tietze as occurring in crystalline schists, but no details of the deposits are given.

The deposits in Italy<sup>56-59</sup> belong apparently to both types—the replacements in marble, and the alteration of serpentines.

#### PYROPHYLLITE DEPOSITS

The most important pyrophyllite deposits in the United States are found along Deep River,<sup>91</sup> in the north central part of Moore and the south central part of Chatham Counties, about 60 miles southwest of Raleigh, N. C. (i.e., they lie in the central part of the state, whereas the talc is in the extreme western part). Some of the deposits have been worked for 75 years. Another deposit, called the Gerhardt, in Randolph County, north of Moore County, has been recently described.

The rocks are volcanic-sedimentary formations made up of slates and tuffs with which are long interbedded bands and lenses of volcanic breccia, flows and ash. Metamorphism has been so complete that it is difficult to distinguish which rock is slate, tuff or flow. Acid, and more basic types of volcanic rocks, exist in the region, and the pyrophyllite is found chiefly in the acid tuff.

#### POLITICAL AND COMMERCIAL CONTROL, PRODUCTION AND CONSUMPTION

Nearly every industrial country has sufficient talc and soapstone resources to supply its own market. There is some foreign trade in particular grades, influenced more by consumer experience and satisfaction with particular products obtained abroad than by the unavailability domestically of those grades. Pyrophyllite is not found so widely, but its applications are not yet sufficiently widely appreciated to have resulted in important foreign trade.

The United States tariff on talc and soapstone, imposed by the Tariff Act of June 18, 1930, is as follows:

Talc, steatite or soapstone, and French chalk, crude and unground,  $\frac{1}{4}$  cent per pound; ground, washed, powdered or pulverized (except toilet preparations) 35 per cent ad valorem; cut or sawed, or in blanks, crayons, cubes, disks, or other forms, 1 cent per lb. Manufactures (except toilet preparations) of which talc, steatite or soapstone, or French chalk is the component material of chief value, wholly or partly finished and not specially provided for—if not decorated, 35 per cent ad valorem; if decorated, 45 per cent ad valorem.

The amount of crude talc produced in the United States has varied between 2150 and 15,625 tons during the last 17 years. The sawed and manufactured talc has varied between 654 and 1528 tons. Soapstone has varied from 16,504 tons to 25,630 tons, during the years when figures have been available. The large volume is in the ground talc and this has varied from a low of 102,413 tons in 1921 to 208,082 tons in 1929. Figures for production by states indicate that the bulk of the ground talc has come from New York, Vermont and California. In recent years the production of North Carolina has been stimulated so that it ranks close to that of California. Imports of ground talc into the United States have varied from a minimum of 11,468 tons in 1921 to 29,949 tons in 1929.

World figures indicate that the United States produces twice as much talc as the next major producer, France, and that Italy, Austria, Manchuria, India, Norway, Canada, and formerly Japan, and in occasional years Spain, are the only countries of which the production exceeds 10,000 tons in a single year. Germany has been a very small producer.

#### MINING AND QUARRYING METHODS

Talc is mined underground at most places but is recovered from open quarries at a number of the smaller operations. All soapstone produced is quarried. Talc bodies, taken as a whole, have every attitude except a



horizontal one. Most are more or less irregular lenses with a dip from 30° to vertical. The width between walls varies from a few feet to over a hundred feet. Hence, no general description of mining methods can be given that is applicable to even a majority of the operations. No talc mine is a large operation. In 1933, the 82,618 short tons produced in New York, the leading producing state, came from at least four mines. The states of Georgia, Maryland, New Jersey, Pennsylvania and Washington together produced only 8093 tons in 1933. For operations of that size, equipment must be simple and inexpensive, and no elaborate development is required.

The Henderson mine, at Madoc, Ont., was laid out with regular levels, and shrinkage stopes are employed. This method is not justified for smaller operations, since too much working capital must be tied up in the stopes. The method employed in New York State, where the veins have a moderate dip, is very simple. The shaft is an incline following the vein. At intervals of 50 ft. vertically (100 to 150 ft. down the dip), a drift is driven in both directions laterally from the shaft, as far as desired, or so that the ore can be worked satisfactorily. These drifts are about 20 by 20, and from them raises up the incline are driven to the next level on 50-ft. centers. Pillars about 30 ft. wide are left between the raises. During the driving of the raises, the blasted ore rolls down the slope to the drift, where it is shoveled into cars, trammed to the shaft, and dumped directly into the skips. The roof stands well, and little or no timbering is required.

The only generality that can be made about drilling and blasting is that the drilling is simple, but that the selection of holes is very important. Presence or absence of slip planes, fractures, irregularities of the many bodies prevent any universal practice. Dynamite is used underground, although black powder is used in soapstone quarries in order to prevent shattering of the stone; and for removing material for crayons, as large blocks as possible are barred down, using a minimum of explosive. New York State operators use a special ammonia gelatin blasting dynamite, either 30 or 40 per cent. In Vermont, a semigelatin is used, called Gel-X-2. Trojan straight powder and 60 per cent Trojan are used in Georgia. Large pieces are broken by secondary blasting or, as at Chester, Vt., by air breakers.

Quarrying of soapstone has been described in detail by Ryan. Where the vein has sufficient width, a wall of soapstone is left on both the hanging-wall and footwall sides of the ledge, to minimize caving. The dimensions of quarries depend upon the width of the vein. The majority are not over 100 ft. wide or long. A few have been carried down to a depth of 200 ft. Where the vein permits, several quarries are opened in line, and a bridge wall 25 to 40 ft. thick is left between adjacent quarries in order to support the walls.

Channeling machines are employed to slice the stone at right angles to the strike of the ledge. These are called "layer cuts." The same machines are used also in making "end cuts" at the hanging-wall and footwall sides of the quarry. Undercutting machines make the bottom slice of each layer. The layers are rectangular prisms of stone 48 in. wide, not including the 2-in. channel cut on each side, 90 in. deep and as long as the quarry is wide. The 90 in. is the depth of each "floor" of the quarry and the 48 in. is the width of each layer.

#### MILLING OF TALC

Talc milling follows a rather general practice. All is dry-ground at the present time and air-separated. Talc for cosmetic purposes is bolted through cloth. In a few talc mills, the fine material must be dried. In the Johnson mill of the Eastern Magnesia Talc Co., the talc that passes through the trommels is conveyed to a drier bin and thence to a coke-fired cylindrical drier. In most mills, however, the talc ore is only damp, so that it needs only the heat of tube mills or Raymond mills to completely dry it.

Fine grinding is done with tube mills, buhr mills, Hardinge mills and Raymond mills. At the mill at Johnson, Vt., the talc is fed to an air separator after leaving the Hardinge mill, and the coarse product goes to a Raymond mill. Raymond mills are used at the grinding plant of the Pacific Coast Talc Co. in Los Angeles. At Chatsworth, Ga.,<sup>32</sup> the talc is fed first to a Williams No. 1 Jumbo Jr. hammer mill, which reduces it to  $\frac{1}{2}$  in. size at the rate of 10 tons per hour. Fine grinding is done with a 50-hp. Williams PW-106, four-roller high-side mill, of which an air separator forms an integral part, the air being blown by a 28-in. clearance fan driven by a 40-hp. motor.

At the mill of the Seaboard Operating Co. at Marriotsville, Md., where most of the product is used for roofing, the talc ore is broken in a hammer mill, then run through a Sturtevant buhrstone mill, operating in series with a battery of screens.

Cosmetic-grade talc is produced in Italy by drying the crude rock, then hand-sorting it into various commercial grades, which are broken with pulverizers, screened, and fine-ground in connection with air separators. Silk bolting cloth is used to screen the finest quality.

Wet grinding, classifying and concentrating is under test at various places, and it is probable that these methods will replace the dry methods rather generally. An urge to adopt wet methods is being given by the wish to avoid silicosis hazards.<sup>94,95</sup> Wet grinding, with pebble mills, in series with wet classifiers and hydroseparators, could replace the present methods. This equipment is already installed at Hemp. N. C., and is being considered elsewhere.

Flotation as a method of concentrating talc has been seriously tested. Tests have been conducted at Rolla, Mo., by the U. S. Bureau of Mines, in cooperation with a leading Vermont producer. A high-grade talc was made, and the low-valued "grit," formerly sold for roofing, was converted to high-grade magnesite, which may have real value.<sup>96</sup> Tests abroad by Tanganyika Concessions<sup>97</sup> have shown that a magnesite-talc rock can be beneficiated by flotation. Other tests have been conducted in Canada.

#### TESTS AND SPECIFICATIONS, MARKETING AND USES

In the trade talc is crudely classified into hard and soft, fibrous and flake; and also by screen sizes and by color. In other cases, it is classified according to the market into which it sells. The actual difference between talcs of various grades and kinds is principally a question of purity. The mineral talc has specific inherent physical and chemical properties. One sample of pure talc can differ little from another except in size of grain, and to a lesser extent in the actual shape of the talc grains or flakes. Most commercial talc on the market, however, is not pure talc but rather a mixture of talc with other minerals. The fibrous talc of New York State, largely used in the manufacture of "cold-water paints," is fibrous because it contains a considerable amount of tremolite. The "gritty" talc of Vermont is a mixture of talc and a carbonate mineral. Many dark colored talcs are mixtures of talc and chlorite or talc and serpentine. Flake and soft talcs are in general white and consist almost entirely only of the mineral talc. Most talcs are sold today on the basis of color, hardness, shape of grain, and on grain size.

In 1931, Bowles and Stoddard\* estimated the proportion of talc sold in various industries in the United States as follows: paint, 48 per cent; paper, 16; roofing, 11; rubber, 11; toilet preparations, 3; foundry facings, 1; ceramics, 1; miscellaneous, 9. In recent years the proportion going into the ceramic industry has increased considerably.

*Uses in Paints.*—Since nearly half of the talc produced goes into the paint industry, it is appropriate to examine the properties of talc that make it a desirable raw material. Talc is essentially an extender, although in some types of paints it may be the principal pigment itself. The loose use of the term adulterant or "filler" is anathema to the paint mixer, and should be deplored. Extenders are added for definite purposes, and the identity of the extender selected is controlled by the properties sought.

Talc is marketed to the paint industry in two forms—as fibrous talc or "asbestine" and as flake talc. Typical examples of the two types were

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\* O. Bowles and B. H. Stoddard: Talc and Soapstone. Mineral Resources of the United States, 1931, U. S. Bur. Mines, pt. II, 100-101.

examined by the writer in order to understand their character. Fibrous talc, or asbestine, is a mixture of tremolite and talc. The fibers, or splinters of tremolite, which make up from 25 to 40 per cent of the mixture have the effect of locking or bonding the paint film, as has been demonstrated recently by Nelson<sup>87</sup> and Broeker.<sup>72,73</sup> The flakes of talc serve as a laminal pigment, the advantage of which has generally become recognized. Asbestine is used largely in cold-water paint or "distemper," as it is called in England, for covering plaster in interior finishes. In this use it is the pigment itself. Here its whiteness gives the necessary hiding power, and the acicular shape of the grains gives bonding. The flake talc is a mixture of talc and a carbonate mineral. The latter probably has no advantage in itself, and is present only because the separation of talc and carbonate minerals is just becoming possible by froth flotation. The carbonate presents an equidimensional grain, which is not desirable. Talc is a very desirable flake pigment, and it is probable that when properly prepared it will be even more widely used than it is today.

*Use in Paper.*—One of the largest uses for talc is in the "loading" of paper. It is used in all grades of paper, from the cheapest to the best. Talc is retained in the cellulose fiber better than clay, and the flaky nature is a desirable property. Only a very white, fine-grained talc can be sold to the paper industry for quality papers. Coarser and off-color material is largely used in the manufacture of roofing paper, and this is the principal outlet for low-grade talc. Here the talc is something more than a filler in the paper. It is placed on the surface as an inert, fireproof and weather-resisting coating. Talc competes with other roofing materials, such as scrap mica, scrap slate, marble dust, etc. The advantage of talc lies in its softness, its flaky nature, its fire-resisting character, etc. The product must sell for a very low price, but since it requires very little preparation the selling of roofing talc is a back log for many producers, and the principal business of many.

*Use in Ceramics.*—Talc as a ceramic product itself has long been known. The product called "lava" or calcined talc has unique and outstanding properties which, though used years ago as tips for gas burners, are becoming even more greatly appreciated today because of the special requirements of the electrical and refractory industries. Because of this unique value of "lava," the material deserves special mention. Diller, Fairchild and Larsen\* studied various raw materials used for the manufacture of lava and came to the following conclusions:

The most essential properties of the best talc known for gas tips are dependent on composition and texture. Even the highest grade of material used for that purpose appears to be not pure talc but a mixture of talc and chlorite. . . . Next to composition, fineness of grain and uniformity of texture is the most essential property of grade.

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\* J. S. Diller, J. G. Fairchild, and E. S. Larsen: High-grade Talc for Gas Burners. *Econ. Geol.* (1920) 15, 672-673.

The conversion of talc to enstatite, clinoenstatite and cristobalite was investigated by Ewell, Bunting and Geller<sup>17</sup> at the U. S. Bureau of Standards, but presumably the material investigated was pure talc. So far as the writer can learn, no one has published in English any results of a study of the physical-chemical reactions in commercial material.

A sample of lava made by the Maryland Lava Co., from its raw material near Dublin, Md., was investigated under the microscope. This material consists of an interlocking mass of splinterlike grains, many of which are curved, together with from 1 to 5 per cent of an isotropic mineral. The grains in a small piece are heavily stained with iron oxide, while a section across the broken face of a larger piece shows heavy iron stain only as a crust on the edges, and occasional spots of iron oxide in the interior. The maximum and minimum indices of refraction of the splintery grains are 1.655 and 1.635, each  $\pm 0.003$ . A few grains show inclined extinction, to a moderate angle, but observations made on most of the grains gave parallel extinction. It is probable that two varieties are present, the indices of one being slightly lower than the other, since the apparent birefringence seen in a thin section of standard thickness is not more than 0.10.

The indices of refraction of enstatite (iron free) are 1.650 and 1.658, and of clinoenstatite (iron free) are 1.651 and 1.660. The observed indices for the mineral, or minerals, in the lava therefore are slightly too low to correspond with the pure magnesium silicate. The presence of iron would raise the indices of the normal metasilicate, therefore presumably no iron has gone into the grains. The isotropic grain has an index of 1.720, and hence is a spinel rather than periclase, MgO.

In the manufacture of lava, great care is used.<sup>81</sup> Blocks free from flaws are selected. The material from Dublin, Md., gives as large blocks as can be obtained at any point in the world, pieces as large as 12-in. cubes having been used. Blanks large enough to form one or many of the desired objects are sawed. These blanks are put in lathes, and skilled machinists turn them to size, cut the threads, drill and tap them according to the requirements of the article. Very intricately tooled articles are made. Threads as fine as 64 to the inch are cut on special units for electrical insulators, refractories and radio parts. The machined articles are then taken to a coal-fired kiln, and placed in it while it is still cold. Electric furnaces are used at one plant. Over a period of many hours the temperature is slowly raised until it reaches 1800° F., at which it is held for about 6 hr. Then the fire is drawn, and the kiln allowed to cool slowly for about 24 hr. Loss by breakage is remarkably low. The finished articles are harder than steel, and the amount of contraction is so slight and uniform that the threads and other dimensions are accurate to 0.0005 inch.

Raw materials for the manufacture of lava blocks are compounded from talc and silicate of soda. The materials are mixed and forced through an extrusion machine, and dried at 600° F. to form a block ready for machining. They are then fired in the same way as is the natural block. The shrinkage is greater, and the loss in firing is said to be higher, but the finished article is as satisfactory, providing proper allowance has been made for the shrinkage. The advantage of the prepared material lies in the color of the finished article. Starting with a white talc and sodium silicate, the finished refractory is a white vitreous body, somewhat similar in appearance to porcelain, rather than the red-brown of lava made from the natural blocks. The American Lava Corporation, of Chattanooga, Tenn., markets such a material under the name of Alsimag.

Considerable research has been carried on in the Soviet Union on the manufacture of refractory bricks from talc and a binder, such as clay, or cement. Talc has been suggested abroad for spark-plug and other high electric insulators.

*Sagger Bodies.*—The largest use of talc in ceramics is in the manufacture of saggars, which are containers to hold and protect green ware during the firing in the kiln. From 2.5 to 15 per cent talc is used in the sagger mix.

*Whiteware.*—Talc has not found a wide use as yet in whiteware, although the National Bureau of Standards has undertaken work investigating such use. R. M. King\* has summarized the merits and failings of talc as a ceramic raw material:

*Advantages*

1. It is a cheap source of MgO that acts as a flux.
2. It imparts to the body high resistance to thermal shock.
3. It has a high specific heat and high resistance to acids.

*Disadvantages*

1. It has a short fusion range, which may impart a short firing range to the ceramic body.
2. Talc of a high iron content may give a body an undesirable color. (Plenty of low-iron talcs are available, or the iron content could be reduced by the use of a ferro filter, for example.)
3. Its use in large proportions would increase costs because, whereas it is a cheap source of MgO, it is an expensive source of SiO<sub>2</sub>.

The use of a tremolitic talc with ground trachyte or rhyolite, in the manufacture of tile in the Los Angeles district, is reported to be growing. The mixture is plastic when finely ground, and is suitable for low-fire

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\* R. M. King: *Ceramic Ind.* (1935) 25, No. 2, 70-71. Quoted in U. S. Bu. Mines Mineral Trade Notes (Nov. 1935) 1, No. 5, 37.

body, such as wall tile. Pyrophyllite has found more general use in whiteware. Sproat\* has summarized the value of pyrophyllite as follows:

A ten-year study of the use of pyrophyllite, both in the factory and the laboratory, has proved that this new ceramic material when properly used in a tile body, will: (1) increase the maturing or firing range of the body, (2) decrease cracking due to thermal shock or moisture expansion or both, (3) decrease fire cracking, (4) decrease the shrinkage with the resulting decreased warpage and (5) decrease die wear.

Some research on the use of pyrophyllite has been conducted in the Soviet Union.

Talc is used in some types of glass, both clear and opal, as it increases the resistance of the glass to chemical attack and mechanical shock.

*Talc as a Filler.*—Talc is used as a filler in so many articles of common use that a complete list would be very long. The most important from a quantitative standpoint is the rubber industry. Talc is used as a filler in rubber goods of all descriptions, and also a large amount is used as a lubricating medium. It is used as a filler in the following: Asbestos shingles, blocks, slabs, etc.; composition floorings; wall plasters; fertilizer compositions; insecticide preparations; linoleum and oilcloth; polishes and soaps; cordage, rope and string; plastics; textile fabrics.

*Talc as an Adsorbent, Lubricant, and Dusting Material.*—The cosmetic and toilet-powder industry is based on talc as the fundamental material, but this market, though high in value, is of little importance to most domestic producers. The quantity used, in comparison with the total for all other consumers of talc, is very small. French and Italian, as well as Manchurian, talc is very widely used by American manufacturers of these materials. The cost of the talc itself is such a small part of the price charged for these package goods generally sold by the ounce, that the cost of the raw material is of no great importance and the duty of 35 per cent ad valorem does not prevent the importation of this product. A French or Italian source probably has a distinct sales appeal to the retail buyer. Few domestic producers, even though they have talc of sufficient whiteness to be acceptable, are equipped to produce or market the grade. It is generally bolted through silk cloth, and sold in 200-lb. bags. One of the California producers markets a grade called "Sierra Cloud."

Talc is used as a lubricant in many industries. It is a constituent of some cup greases, it is used on wire nails used in automatic box-nailing machines, for dies of various types, in pneumatic tires to prevent pinching of the tube, in rubber gloves, boots and shoes. It is an absorbent for drying oily leathers, for nitroglycerin in some explosives, in toilet soaps, lotions and face creams. Talc is used as a dusting agent to coat foundry molds, bottle molds to prevent the adhering of the glass, in cork molds,

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\* I. E. Sproat: Use of Pyrophyllite in Wall-tile Bodies. *Jnl. Amer. Ceramic Soc.* (May, 1936) 19, 138.

*Quotations on Talc*

Grade	1922	1929	1932	1934	1936	
					Grade	Quotations
Vermont, 20-50 mesh roofing, in bulk.....	\$7.50- 8.50			In bags \$16 00-18.00	Vermont: In bulk, f.o.b. works. 99 per cent through 200 mesh	\$9.00; in bags, \$1 per ton extra
Vermont 150-200 mesh, bulk....	8.00-16.00				97 to 98 per cent through 200 mesh	\$8.50; in bags, \$1 per ton extra
New York, fibrous 150-300, in bags.....	14.75-18.00		\$20.00-25.00 <sup>a</sup>	20 00-25 00 <sup>a</sup>	New York: In bulk, f.o.b. works. 325 mesh	\$12.00-\$15.00
California, toilet powder grade, in bags.....	16.00-20.00		18 00-25.00 <sup>a</sup> 22 00-27.50 <sup>a</sup>	18 00-25.00 <sup>a</sup> 23 00-23 00 <sup>a</sup>	Georgia. In bulk, f.o.b. works. 98 per cent through 200 mesh	
Canadian, imported in bags . . .					Gray	\$6.00; in bags, \$1 per ton extra
					White	\$8.00; in bags, \$1 per ton extra
French, ex dock, in bags (high grade)....	40.00-45.00		35.00-40.00	45.00-60.00	Virginia: in bulk, f.o.b. works. 200 mesh 325 mesh	\$4.75 to \$5.00 \$6.20 to \$7.00
Italian, ex dock, in bags . . . . .	40.00-55.00		48.50-55.00	70.00-85 00	Crude	\$4.00
Average value of ground talc . .	\$11.90	\$11.50	\$11.00	\$10.40		

<sup>a</sup> Delivered.



on linoleum and for coating candy molds. It is used as a packing material for eggs and fruits. Talc is used as an abrasive in spite of its extreme softness. It goes into the various compounds for removing "traffic film" from automobile finishes where a harsher abrasive would scratch the lacquered surface, and for wood polishes and for scouring foodstuffs, such as barley, beans, coffee, corn, peanuts, et cetera.

*Soapstone.*—Forty per cent of the output of soapstone goes into the smelting furnaces of sulphite (Kraft) pulp mills. As a material of construction, it has wide application. Soapstone is used for acidproof flooring, table tops, sinks, tanks, vats, laundry tubs, etc. Because of its high electrical resistance, it is suitable for switchboards and as other insulating media. Because of its long retention of heat, and fracture resistance under repeated heating, it is used in fireless cookers, ovens, etc. Small pieces of soapstone are used for crayons for marking cloth, metal, glass, etc. It is one material that will make a mark on hot or cold metal that is visible when the metal is cooled or heated. These crayons are the principal production of some mills. Those most used are  $\frac{1}{2}$  by  $\frac{3}{16}$  by 5 in. They are sawed by circular saws from blocks of suitable quality and size.

#### PRICE HISTORY

So many grades of talc are offered that the quotations vary widely. The quotations in the accompanying table, taken from published market reports in 1922, 1929, 1932, 1934 and 1936, show the trends during the predepression, the depression and recovery periods.

It is apparent that although the quoted selling prices for certain grades of talc have been on the increase, the average value of ground talc sold in the United States has been on the decline. There has been such keen competition in the lower grades of talc that many sales have been made at lower prices than those quoted. Much roofing talc has sold as low as \$4 per ton, f.o.b. shipping point.

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## CHAPTER XLVII

### TITANIUM

BY FRANK L. HESS\* AND JOSEPH L. GILLSON,† MEMBERS A.I.M.E.

**E**XCEPT iron, aluminum and magnesium, titanium is more plentiful than all of the metals that we call "common" added together, but it holds to oxygen, or lacking it, to other elements with such tenacity that it has been obtained practically pure only with great difficulty and, until recently, when separated, it had little use except as a curiosity. Added to the fact that it has been so difficult to separate from its chemical partners, its minerals have shown no characteristics that made their usefulness readily apparent. These two items have made titanium and its minerals late comers in the world's general markets—in fact, they are only now arriving. Titanium has come into its own as a real article of commerce mostly because it was found that one of the blackest of minerals, ilmenite, could be used as a source of the whitest of pigments.

#### MINERALS

A considerable number of minerals carry titanium, but those of commercial importance are few. Their names and some of their properties are as follows:

*Rutile*.—Rutile [ $\text{TiO}_2$  (60 per cent Ti)] crystallizes tetragonally, has two fair cleavages, a chonchoidal fracture and a metallic-adamantine luster. Its color is reddish brown, but may be yellow, yellowish brown or nearly black, and occasionally white or green. The lighter colored rutile is translucent, but even microscopic particles of the darker colors are nearly opaque. Its hardness is 6 to 6.5; specific gravity, 4.242 to 4.249. It is insoluble in acids, but readily attacked by fused alkali-metal salts, and is then soluble. Iron to the extent of 1 or 2 per cent, or even 3 per cent, is often present.

Some rutiles with the higher iron content are jet black and are known as *nigrine*. One from St. Peters Dome, Colorado, carries 3.77 per cent  $\text{Fe}_2\text{O}_3$ .

*Octahedrite*.—Octahedrite, sometimes called "anatase," is also  $\text{TiO}_2$ . Its crystallization is tetragonal but usually in octahedrons, which may be

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very much modified. Hardness is 5.5 to 6; specific gravity, 3.82 to 3.95; color, brown, indigo blue, black; otherwise like rutile.

*Brookite*.—Brookite is  $\text{TiO}_2$ . It crystallizes in orthorhombic forms, usually in thin tabular crystals. Stout crystals have been called "arkan-site." Hardness, 5.5 to 6; specific gravity 3.87 to 4.01; colors and other characteristics as in rutile.

*Ilmenite*.—Ilmenite is ferrous titanate ( $\text{FeO} \cdot \text{TiO}_2$ ). It carries 52.7 per cent  $\text{TiO}_2$  or 31.6 per cent Ti. It is seldom found in distinct crystals, but occasionally grows in rhombohedral forms. It is opaque black with a submetallic luster, conchoidal fracture and black or dark brown streak and powder. Hardness is 5 to 6; specific gravity, 4.5 to 5. Dana says that the ratio of titanium to iron varies widely, but the variation probably is due either to molecular or to microscopic intergrowths of arizonite, geikielite, pyrophanite, hematite and magnetite. Ilmenite slowly decomposes in hot hydrochloric or sulphuric acid.

*Arizonite*.—Arizonite, ferric titanate ( $\text{Fe}_2\text{O}_3 \cdot 3\text{TiO}_2$ ; 60 per cent  $\text{TiO}_2$ , 40 per cent  $\text{Fe}_2\text{O}_3$ ), though not known to have been recognized heretofore, seems to form the larger part of some sands classed as ilmenite, particularly those of Quilon and Cape Dakar (Senegal). It is possibly monoclinic. Its color is dark steel gray with submetallic luster; streak, brown; transparent, deep red in thin sections. The  $\text{Cr}_2\text{O}_3$  in ilmenite may take the place of  $\text{Fe}_2\text{O}_3$  in included arizonite molecules.

Other titanium minerals that may conceivably become useful are "sphene" or "titanite" ( $\text{CaO} \cdot \text{TiO}_2 \cdot \text{SiO}_2$ ) and "perovskite" ( $\text{CaO} \cdot \text{TiO}_2$ ), but of neither mineral are known bodies large enough to allow profitable exploitation.

Like arizonite, two other titanium minerals are known separately only as mineralogical curiosities, and are worthy of mention only because, as already noted, they form intergrowths with ilmenite. These are *geikielite*, the magnesium titanate ( $\text{MgO} \cdot \text{TiO}_2$ ) and *pyrophanite*, the manganese titanate ( $\text{MnO} \cdot \text{TiO}_2$ ).

## GEOLOGY

All of the titanium minerals just mentioned are of deep-seated origin. Rutile in minute crystals and small quantity is found in some of the dark micas, in larger crystals in quartz veins closely related to pegmatites, and in schists into which siliceous solutions have carried the titanium. The known large deposits are in pegmatites or in very closely related basic rocks such as krageröite, a rutile plagioclase rock. Apatite and albite are common associates.

Ilmenite in flat, imperfect crystals is found here and there in quartz veins, but not in commercial amounts. In large deposits it most commonly occurs as plates intergrown along the cleavage planes of magnetite,

but the plates may be microscopic in size. Singewald (ref. 44,\* p. 141 et seq.), after considerable study, concluded that some magnetites contained titanium as an integral part of the magnetite molecule, but it seems possible that the plates of ilmenite may be too small to be seen even with a microscope. In some magnetites the ilmenite is in comparatively large particles and is readily separated magnetically.

Ilmenite also occurs as an intergrowth with hematite, in both the massive and the specular varieties. Ilmenite magnetite and ilmenite hematite in large bodies are found only with gabbros, anorthosites and similar rocks.

Pure ilmenite in place is known in rich minable bodies only in the peculiar rock known as nelsonite, in which ilmenite and apatite, or rutile and apatite, are the essential minerals. Phlogopite, quartz, pyrite, pyroxene and hornblende may be accessories. In some nelsonites pyroxene and/or hornblende may be the principal minerals with apatite. The origin of nelsonite bodies is not altogether clear. They have the form of dikes and seem to be of igneous origin, yet a few seem to be somewhat crustified. Apatite appears to have been a primary mineral in the bodies and to have been replaced by ilmenite or rutile until in some places only small rounded pellets of apatite are left.

By far the greatest quantity of ilmenite, however, is in small dispersed grains or plates: (1) in the highly metamorphosed deep-seated rocks such as the schists and gneisses, (2) in the andesites and more basic lavas, and (3) in the gabbros and more basic granular rocks. In both lavas and granular rocks, however, magnetite is probably always present also in relatively large amounts.

Both rutile and ilmenite are extremely resistant to weathering, so that as the rocks are weathered and eroded the two titanium minerals and quartz accumulate in the "black sands" of streams and beaches. These placer deposits are the easiest and cheapest to work, and furnish the great bulk of the world's titanium. Locally stream gravels furnish a limited supply of rutile, but the vastly greater amount of ilmenite than of rutile in the world is shown by the paucity of beach deposits carrying a notable quantity of rutile. The disparity in their quantities is probably in the ratio of several thousand parts of ilmenite to one of rutile.

The concentration on some beaches is remarkably high, those at Travancore, India, and New South Wales, Australia, showing a concentration of ilmenite, rutile and zircon to the extent of 75 per cent or even more of the black sand.<sup>51</sup> The content of the concentrates, of course, varies with the heavy, hard minerals of the rocks of the drainage area. It is impossible to get high ilmenite concentrations where large quantities of garnet are fed into the sea, as at Nome, Alaska, or where the ilmenite is mixed with large quantities of magnetite from basaltic or other lavas.

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\* References are at the end of the chapter.



## DISTRIBUTION OF DEPOSITS

## RUTILE

The largest rutile deposit known, that at Roseland, Va., is in a broad pegmatitic replacement of a peculiar aplite\* composed wholly of a sodic plagioclase into which interfingers a small quantity of potash feldspar (antiperthitic structure). The aplite is intruded into the Lovingsston granite gneiss. After the aplite had solidified it was broken with innumerable cracks and some considerable faults. Along these openings solutions rose, which deposited grains of clinozoisite, blue quartz, pyroxene, hornblende, rutile and ilmenite by replacement of the aplite. Where the cracks were wider or crossed each other so that solutions flowed more freely, the replacements were larger and rutile formed in masses, some of which weighed several pounds each. As in most pegmatitic or other replacement deposits, the proportions of the minerals vary from place to place.

At Roseland great quantities of oligoclase,† some in large lumps 6 to 12 in. across, apparently thus formed a body several hundred feet broad, but the shape cannot be seen because of the soil covering. Along the sides are masses of later hypersthene (now largely changed to hornblende), and blue quartz mixed with the oligoclase. Rutile and ilmenite in comparatively small grains follow cracks through the pegmatite, but the ilmenite is mostly with the hypersthene and hornblende. There is also considerable white apatite. Much of the rock mined is said to average 4 to 5 per cent each of rutile and ilmenite. The deposit is owned and operated by the American Rutile Co., a subsidiary of the Metal and Thermit Corporation of New York. The deposit has been worked as a quarry since about the beginning of the century.

The rock is crushed and separated on shaking tables, thus making a concentrate which in the long run seems to give about equal quantities of rutile and ilmenite. These concentrates are then run under a Wilfley magnetic separator, which lifts out the ilmenite.

The rutile concentrates carry from 92.5 to 98 per cent  $\text{TiO}_2$ . An analysis of ilmenite concentrates is given in Table 2.

The old mill had a capacity of 500 to 600 tons of rutile per year, but a newer one is unauthoritatively reported to have a capacity of 1800 to 2000 tons per year, and both mills can be used. The feed is now said to average about 3 per cent each of rutile and ilmenite.

A group of greatly decayed pegmatites carrying dull red rutile intergrown with ilmenite and white and green apatite has been prospected at

\* "Aplite" is used here in the broad sense of a granular igneous rock free from dark minerals. The rock is called syenite, "but andesine-anorthosite would define it more exactly."<sup>60</sup>

† Clarence S. Ross has determined the feldspar as varying from Ab75 to Ab79 with antiperthitic microcline.<sup>43</sup>

considerable expense on the Nuckols farm, between Peers and Johnson's Spring in Goochland County, and on the Bowe farm, near Gouldin, Hanover County, Virginia, but the deposits have proved to be too small<sup>21</sup> for profitable mining.

At Magnet Cove, Hot Spring County, Arkansas, pegmatitic phases of the nepheline syenite carry dark rutile, octahedrite and brookite. Most of the pegmatite bodies are small and none of the masses is large enough or rich enough to be mined profitably, but considerable quantities of the syenite have decayed and have been washed away, leaving the unweatherable  $\text{TiO}_2$  minerals in the detritus, which has been worked at Malvern by two companies.

Somewhat similar to the Magnet Cove occurrence is that on Shooting Creek, North Carolina, where narrow veins that were wholly quartz where seen but probably carry feldspars in their wider parts, have set free considerable quantities of rutile. The mile-broad valley has been rather extensively drilled but no actual mining has taken place. Additional but smaller occurrences of rutile have been found through the length of the Appalachians.

On Kragerö Island, about 75 miles northeast of Oslo, Norway, rutile in dark rounded grains is found in krageröite. A few tons of rutile has been produced annually for many years.

At St. Urbain, a few miles north of Baie St. Paul, Quebec, an ilmenite-hematite deposit carries as much as 6 per cent of red rutile with sapphirine, plagioclase and apatite, in bands and spots as much as 2 to 3 ft. wide. Anorthosite cutting the ilmenite hematite carries from 2 or 3 per cent to as much as 15 per cent of rutile, some ilmenite and biotite but no sapphirine.<sup>49</sup> The titanium minerals and hematite are replacements of the anorthosite country rock.<sup>18</sup> At one time the General Electric Co. separated some rutile from the aggregate.

In Brazil, rutile is found in the conglomerates and in the diamond-bearing gravels, especially near Pyrenopolis, in the southern part of the State of Goyaz, and in adjoining parts of Matto Grosso. Pieces range in size from 3 to 30 mm. in diameter and average perhaps 5 mm. Some of the pieces are of gem quality. Concentrates obtained by hand washing carry 95 to 98 per cent  $\text{TiO}_2$  and are shipped to New York and London.

Near Bom Jardim, Livramento, Avuruoca, Turvo and Lima Duarte in the Rio Grande basin of southern Minas Geraes larger quantities of rutile, which is, however, intergrown with ilmenite, are found in a conglomerate cemented by limonite. The rutile is derived from pegmatites, mica schist, and quartzite country rock. The rutile content of the conglomerate is variable but samples gave from 3.11 to 8.74 kg. per cubic meter, and material carrying 7 kg. per cubic meter is considered rich. Owing to the intergrown ilmenite the concentrates carry only 70 to

85 per cent  $\text{TiO}_2$ . The fragments are somewhat larger than those from Goyaz and Matto Grosso, and range from 10 to 50 mm. in diameter.<sup>1</sup>

In Diamantina, Brazil, the diamond-bearing gravels carry some rutile. Brazilian rutile is now coming to this country in considerable quantity. In 1935, there were received in the United States 212 short tons and in the following year 268 short tons of Brazilian rutile. In 1936 prices ranged from 2 to  $3\frac{1}{2}$ ¢ per pound.

Mexico has furnished specimens of rutile-bearing rock from the coffee plantation San Francisco at Pluma Hidalgo, Oaxaca, that look as if they might have originated at Roseland, Va. Nothing is known of the extent of the deposit. Red rutile in white albite (?) has also been received from some carefully concealed place in Mexico, supposedly south of Duquesne, Arizona.

In Madagascar, rutile is found in narrow quartz veinlets cutting mica schists west of Ambatofinandrahana and some production was made from the deposits during the Great War.<sup>32a</sup>

In Sierra Leone, on Mabole River between Robonko and Romakut, 2 and 4 miles respectively above its junction with Little Scarcies River, gravels carry 7 lb. of rutile in pieces of  $\frac{1}{2}$  in. or larger in diameter associated with ilmenite, zircon and a little corundum. How much finer rutile there may be is unknown.<sup>41</sup>

In French Congo important deposits of rutile are said to exist at N'Dacire at the contact of quartzite and granulite.<sup>11</sup>

Rutile has been reported in the schists of upper Dahomey and the Ivory Coast<sup>32a</sup> and is also reported in French Cameroun.<sup>32b</sup>

A dark rutile with a submetallic luster, from some undisclosed point in northwestern Africa, is now being offered in the American market. It is evidently from a pegmatitic deposit.

In South Australia, rutile was formerly mined in small quantity from a pegmatite 15 miles east of Gawler. Other occurrences noted are: a kaolinized dike in the hundred of Talunga, 6 miles north of Blumberg;<sup>10</sup> "in matrix" at Mt. Crawford, hundred of Para Wirra;<sup>17,52</sup> hundred of Yankalilla;<sup>26</sup> hundred of Myponga,  $\frac{1}{2}$  mile northwest of Mt. Cone.<sup>53</sup>

The zircon-ilmenite-rutile placers of the northern coast of New South Wales probably will become active producers of rutile as a by-product of zircon.

#### ILMENITE

Although ilmenite, at least in small quantity, is found practically wherever igneous rocks are known, pure massive ilmenite is not known in large primary deposits, though many ilmenite-magnetite and ilmenite-hematite bodies have been found with gabbroic and anorthositic rocks. One of the largest, and the purest so far described, is the Blaafjeldit

ilmenite-magnetite deposit of the Titan Company at Jossing Fjord, in Sogndal south of Egersund, Norway. After concentration the ore carries about 42 per cent  $\text{TiO}_2$ ,<sup>27</sup> and part of it is said to carry 45 per cent  $\text{TiO}_2$ . The deposit is estimated to contain 30,000,000 metric tons of such ilmenite to a depth of 100 meters. For some years this ore was shipped to England to be smelted for iron, and a short railroad was built to carry the product to the coast. Since about 1920 it has been worked for the production of titanium white and it was on this deposit that the titanium-white industry was started. It is now controlled by the National Lead Co. of New York. The Laxedals deposit close by carries 35 per cent  $\text{TiO}_2$  and contains about 250,000 metric tons of ilmenite magnetite. In 1935 the United States imported 22,472 tons of ilmenite concentrates from Norway, presumably from the Blaafieldit deposit.

In the United States the largest deposits of titaniferous iron ores are those of the Adirondacks, where bodies of ilmenite magnetite are enclosed in country rocks of anorthosites and dark gabbro or norite. The ore, which carries from 7 to 23 per cent  $\text{TiO}_2$ , is too lean, however, to compete with the ilmenites now being marketed,<sup>31,39</sup> although it is claimed that 100,000,000 tons of ore has been proved.<sup>3</sup> Some of the ore has been used for making ferrotitanium, but none is known to have been mined within recent years. Singewald has discussed the magnetic separation of the ores and has found it only moderately promising (ref. 44, pp. 47-76).

On Chugwater Creek, 9 miles west of Iron Mountain station, Wyo., is a mass of solid ilmenite magnetite 100 to 200 ft. wide in labradorite rock, extending  $\frac{1}{2}$  mile to the north and to the south (refs. 5; 34; 44, pp. 111-125). Analyses show from about 19.5 to 49.5 per cent  $\text{TiO}_2$  but most of them give 22 to 23 per cent  $\text{TiO}_2$ . The highest analysis was not duplicated.

There are several smaller dike-like bodies in the vicinity and 6 miles southwest a body 1600 ft. long has a maximum width of 80 ft. Minor masses are said to be found from Horse Creek to Sibylee Creek, a distance of 20 miles. The Colorado Fuel and Iron Co. at one time shipped a considerable quantity of the ore to its blast furnaces at Pueblo, Colo. The ore does not lend itself well to magnetic concentration.

In California, ilmenite-magnetite mixtures are found in the mountains about 8 miles (16 miles by road and trail) southeast of Acton, near Mill and Fuller Creeks, in secs. 14 and 23, T.3N., R.12W.; and in T.3N., R.13W., S. B. B. & M. The titanium oxide content is said to range from 11 to 25 per cent. Other deposits are said to be found in sec. 36, T.6N., R.14W., 10 miles north of Acton, and still others near Lang and Russ.<sup>12</sup>

Smaller deposits of titaniferous magnetites are known in Fremont (ref. 44, pp. 128-135) and Boulder<sup>28</sup> Counties, Colorado; Minnesota,<sup>9</sup> Rhode Island;<sup>29</sup> North Carolina;<sup>40</sup> and elsewhere.

In Amherst and Nelson Counties, Virginia, which lie on either side of Piney River, there are nelsonite dikes large enough to be of real value as producers of ilmenite. As now known, much the largest of the dikes is that in Amherst County, across the river from Piney River station on the V. B. R. Railroad (Fig. 1). It is on the property of the Southern Mineral Products Co., a subsidiary of the Vanadium Corporation of America. The body outcrops 70 or 80 ft. high for 350 ft. along the river, in a bluff at the edge of an old terrace. It enlarges to a surface width of 400 ft. and carries its width for about 1300 ft. to the southwest, then

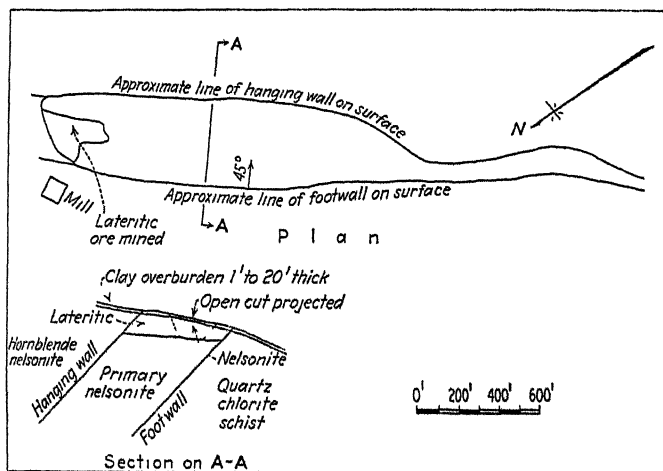


FIG. 1.—PLAN AND CROSS SECTION OF NELSONITE DIKE WORKED BY SOUTHERN MINERAL PRODUCTS CORPORATION, PINEY RIVER, VIRGINIA. (AFTER F. F. KETT.)

in the next 600 ft. it tapers down to somewhat less than 100 ft. and a tail 60 to 100 ft. wide extends for more than 1000 ft. farther.

The nelsonite is enclosed in a biotite-schist phase of the Lovingson granite gneiss and extends out from the edge of the aplite. It dips with the schist about  $45^{\circ}$  SE., so that the surface exposed is much wider than the actual thickness of the dike, which is about 283 ft. where the outcrop is 400 ft. across, and the tail is 28 to 71 ft. thick. The dike has been drilled to a depth of about 400 ft. and appears to continue below that point.

For about 75 ft. below the surface, the nelsonite is a rather rusty rock, so soft that it can be crushed in the hand. Beyond that depth it hardens, the ilmenite is a clean black, the apatite is comparatively clear, and there is enough chlorite to give a greenish tint to the rock. A little pyrite occurs along cracks.

Only the decomposed rock is now being quarried, and a face about 80 ft. high is being worked. The rock contains 18.5 per cent  $\text{TiO}_2$ , and if the concentrates represented in analyses 1 and 2 in Table 2 are typical the rock should yield about 42.5 per cent of concentrates. The remainder

is almost wholly apatite. It is said that below the weathered zone the rock is quite as amenable to treatment as is that above.

Considering that the deposit extends to a depth of 400 ft., and that the specific gravity is 3.89, nearly 24,000,000 tons of nelsonite containing more than 4,400,000 tons of  $\text{TiO}_2$  is indicated. Of course, where so few data are at hand, the figures must be treated with due reserve.

The manufacture of titanium white from this rock is still in the experimental stage but for a number of years large quantities of calcium monophosphate (used in baking powder) have been manufactured from the apatite.

About  $1\frac{3}{4}$  miles down Tye River from Massies Mills, the National Lead Co. has a hard, vertical nelsonite dike, 60 ft. wide and 600 ft. long, on the side of a hill sloping toward the river, and the Southern Mineral Products Co. has a smaller hard dike of about the same width but shorter, on a hillside sloping toward Hat Creek. Two miles farther up Hat Creek, the Burgess Titanium Co. has a similar dike not thoroughly prospected, but thought to be 40 ft. thick and of considerable length. These deposits are said to carry 30 per cent  $\text{TiO}_2$  and probably contain at least 2,000,000 tons of rock, or 600,000 tons of titanium oxide.

In the two counties there are also many smaller nelsonite dikes, which, should the titanium-white industry continue to expand in the area, may contribute an appreciable quantity through operators with small capital.\*

The American Rutile Co. probably will produce from 1000 to 2000 tons of ilmenite per year from its rutile plant at Roseland. An analysis of the ilmenite concentrate may be compared with those of other ilmenite concentrates in Table 2.

Canada has numerous deposits of ilmenite magnetite and ilmenite hematite. The best known are those at St. Urbain, near Baie St. Paul.<sup>36</sup> Several thousands of tons of ore carrying 35 to 40 per cent  $\text{TiO}_2$  have been shipped to Niagara Falls, N. Y., for the manufacture of ferrocenone titanium. Gillson<sup>18</sup> notes that the ores are replacements. Somewhat similar deposits, but without rutile, are known at Ivry, Terrebonne County. It is said that the ores carry 18 to 25 per cent  $\text{TiO}_2$  and that some have been shipped to Niagara Falls, N. Y.<sup>13</sup> Other known Canadian deposits include one in Alberta; a number of bodies in the Chicoutimi district, Quebec,<sup>42</sup> carrying 12.40 to 22.42 per cent  $\text{TiO}_2$ ; masses 10 to 61 ft. wide, carrying 17 to 21 per cent  $\text{TiO}_2$ , besides larger but leaner bodies near Seine Bay, in the Rainy Lake region; with apparently less important deposits in Renfrew, Hastings and Haliburton Counties, at Newboro (carrying 9.8 per cent  $\text{TiO}_2$  and once smelted for iron<sup>19</sup>), and in the Nipissing district, Ontario.<sup>22</sup>

Europe, aside from the Scandinavian peninsula, to which reference has already been made, is not rich in titanium deposits. Russia has a

\* Many of these bodies are shown on a map by T. L. Watson and Stephen Taber.<sup>50</sup>

number, the largest of which seems to be in the Ilmen Mountains, a branch of the Urals, where a deposit is said to contain 400,000,000 tons. The ores carry 15 per cent  $\text{TiO}_2$  and when magnetically concentrated give a product with 43 per cent  $\text{TiO}_2$  and an iron concentrate carrying 65 per cent Fe.<sup>2</sup> A large deposit is reported also near Khibine, on the Kola Peninsula, and another near Gatskavo, in the Ukraine.<sup>14</sup>

In Africa ilmenitic magnetite deposits have been reported from Madagascar<sup>8,33</sup> (10 to 40 per cent  $\text{TiO}_2$ ); the Arabian desert of Egypt;<sup>45</sup> Dahomey,<sup>7</sup> and Sierra Leone.<sup>30</sup> Others will probably be found with further prospecting.

In Asia deposits in Korea<sup>23</sup> and Manchuria<sup>46</sup> have been described, and others have been reported in French Indo-China. An otherwise unimportant deposit at Kancharia, India, carrying 43 per cent  $\text{TiO}_2$ , may be worthy of mention because it has been smelted by the natives for iron.<sup>20</sup>

A few hundred tons of ilmenite ore are mined in Portugal each year.

#### BEACH DEPOSITS

Black sands on ocean beaches now supply about half of the ilmenite consumed. The most productive beach in the world lies on the west side of the south tip of India, within the native state of Travancore. There are two strips of the Travancore coast that are productive; one in the southern part of the state at Manavalakurichi ( $77^\circ 18' \text{E. long.}, 8^\circ 8' \text{N. lat.}$ ) and the other 80 miles north, and 4 miles north of the well-known town of Quilon.

The beach at Manavalakurichi (known to European residents as "M. K.") has been worked since 1911, but until about 1924 monazite was the only mineral sought, and the ilmenite was of no value. Up to 1934, nearly all of the production came from this beach, but the reserve there has been largely depleted. The productive section is about 6000 ft. long, arcuate in form, extending westward from a rocky headland at Muttum toward the headland at Colachel, in the lee of which the steamers lie, while being loaded from dugout canoes. Part of the sand has been "harvested" from "washings"—i.e., natural concentrations on the beach front worked over by the waves—and the rest has been mined from buried highly concentrated seams formed at earlier stages in the development of the beach. The crude sand worked carries 50 to 70 per cent of ilmenite, although the 6 to 8 ft. of overburden removed from above the buried seams has been much lower. The sand is gathered by means of a momiti, a hoelike tool, universally used in agriculture in India. The sand is placed in baskets and transported on the heads of "boys" to cars for haulage to the concentrating plants. At one of these, wet tables are used to remove the bulk of the quartz. The wet sand from the beach

or tables is dried in the sun, and further concentrated by dry tables and by magnetic separators, to eliminate the zircon, monazite, rutile, garnet and other minerals. The zircon and monazite are themselves separated and concentrated to marketable by-products.

Travancore Minerals Company, Ltd., has worked the beach front while another firm, Hopkins-Williams Travancore Ltd., has developed buried seams of black sand behind the present beach.

The northern productive beach extends from a short distance south of Neendakara Inlet to Kayankulam Bar, about 15 miles to the north. Neendakara Inlet is 4 miles north of Quilon, and when visited by Marco Polo was one of the busiest ports in the world. Travancore Minerals Company, Ltd., has erected a large concentrating plant at the village of Kovilthatam, about 8 miles north of Quilon, and is now producing its major tonnage there. A native firm, F. X. Periera and Sons, purchased private land on the dunes back from the beach north of Kovilthatam, and is producing on a moderate scale.

The production of ilmenite in Travancore has risen from 641 long tons in 1924 to 129,090 long tons in 1935, and was even larger in 1936. The total production has exceeded 700,000 long tons. A large part of the recent large production has not been consumed, but has been used to build up stocks in the United States. As shown in Table 2, the M. K. ilmenite carries 54 per cent  $\text{TiO}_2$ , whereas that from Quilon runs from 59+ to nearly 61 per cent.

Brazil has large beach deposits of mixed ilmenite, zircon, monazite and other heavy minerals along the coast of Espirito Santo, Rio de Janeiro and Bahia. Exports of 6361 tons were reached in 1929 but ceased suddenly. It is surmised that the competition of the Travancore deposits was too strong. However, there is undoubtedly a very large quantity of ilmenite on the Brazilian beaches that in time will be concentrated and exported.

In Australia, Titanium Pigments Ltd. (Melbourne) has produced ilmenite at Naracoopa, at the mouth of Fraser River on King Island, 60 miles north of Tasmania in Bass Strait. Besides ilmenite, the sand carries cassiterite, monazite and gold. The black sand is about 8 ft. thick and the surface is 6 ft. above high-water level. Magnetic concentrates are said to carry about 45 per cent  $\text{TiO}_2$ .<sup>24</sup> In 1933, this company produced 559 tons of concentrates. Along the central eastern coast of Australia extensive and very concentrated beach sands extend from Shoalhaven River, N.S.W., well toward Brisbane, Queensland. They have been worked intermittently for 35 years for tin, gold and platinum. The largest deposits are north of Port Macquarie. Along the coast are old beach remnants raised a few feet above the present beach.

Microscopic counts of the natural concentrates, apparently somewhat further cleaned, gave: rutile, 7 per cent (Ballina) to 18 per cent



(Iluka); ilmenite, 14 per cent (Berrie Point) to 43 per cent (Collaroy); zircon, 40 per cent (Collaroy) to 75 per cent (Berrie Point).<sup>51</sup>

The Zircon Mining Co., a subsidiary of Titanium Alloy Manufacturing Co., of Niagara Falls, N. Y., is working part of the beach for zircon at Yamba, at the mouth of the Clarence River.<sup>16</sup> Metals Resources, Ltd. (Sydney), has a lease near Brunswick Heads, and Zircon Rutile, Ltd. (Melbourne), a lease near Byron Bay. The International Titanium Co., of Toronto, Canada, holds sands said to be rich in rutile, on Byron Bay on the Queensland end of the beach,<sup>38</sup> but so far has shipped only zircon. At the time of writing, only the Zircon Mining Co. seems to be active.

The United States at one time produced some thousands of tons of ilmenite with considerable zircon and a few tons of rutile from the east Florida coast, 4 miles south of Jacksonville beach, but the deposit was exhausted and now probably no other American sands can compete in the world's market.

In the Federated Malay States ilmenite forms a considerable part of the *amang* (heavy tailings) accompanying cassiterite in the placers, and at mines where magnetic separation is used the concentrate is ready to ship. Some concentrates also contain zircon. During the first nine months of 1936, mines in these States exported 9625 short tons to Europe. From a part survey of the field, it was estimated that 241,000 long tons was available.<sup>15</sup> As most of the tin mining is in Perak, a large part of the *amang* comes from there. As the ilmenite is waste, it can be loaded on shipboard at Port Swettenham at \$1 to \$2 per long ton.<sup>37</sup>

Japan has great quantities of titaniferous magnetite sands, derived from her many and widespread lavas and from old metamorphic rocks. The sands are found on most beaches of Japan proper (Hondu), Kyu-shu and Hokkaido. The most concentrated sands are on the east coast of Aomori and Iwate prefectures, Hondu, where thick layers of magnetic sand are cemented with ferric mud. The deposits are estimated officially to contain 10,000,000,000 tons,<sup>35</sup> carrying as mined 20 to 30 per cent Fe and 8 to 12 per cent  $\text{TiO}_2$  and 0.6 per cent  $\text{V}_2\text{O}_5$ . The sand can be concentrated to 60 to 65 per cent  $\text{TiO}_2$ . Kuji, 100 miles from the north tip of the island, is in the middle of a strip 70 miles long and 20 miles wide, which has been raised above sea level and on which the iron sands average 18 ft. in thickness, with a minimum of 200,000,000 tons. An official estimate was 150,000,000 tons in 22 sq. km. (8.6 sq. miles).<sup>25</sup> Several companies are starting operations for making titanium white and two are producing ferroc carbon titanium (15 to 20 per cent Ti, 6 to 9 per cent C) by reduction in arc furnaces.

Argentina has considerable beach deposits, which extend southward from Necochea to and through the Straits of Magellan.<sup>48</sup> The sands are said to carry: magnetic iron sand, 29.22 per cent; ilmenite, 24.76 per cent; ferruginous black sand, nonmagnetic, 27.89 per cent.

## POLITICAL AND COMMERCIAL CONTROL AND WORLD PRODUCTION

As indicated in the preceding descriptions, the control of much the larger part of the ilmenite is in British governmental and commercial hands. From its own deposits the United States probably is on a par with Norway. In French territory none but French companies may operate, and exports are carefully watched; the same is true of Russia. No duties are levied in the United States on titanium concentrates.

The world's production of titanium minerals is shown in Table 1.

TABLE 1.—*World Production of Titanium Minerals, 1931–1935<sup>a</sup>*

METRIC TONS

Mineral and Country	1931	1932	1933	1934	1935
<b>Ilmenite:</b>					
Australia (Tasmania) . .			559		<sup>b</sup>
Canada (Quebec) . . . . .	1,369			1,835	2,076
Egypt . . . . .		487		164	183
French West Africa (Senegal) <sup>c</sup> . . . . .	370		160	540	1,250
British India (Travancore) . . . . .	36,746	50,856	53,830	76,858	129,090
Federated Malay States <sup>c</sup> . . . . .	<sup>b</sup>	<sup>b</sup>	204	51	2,540
Norway . . . . .	5,000	13,481	23,213	26,306	37,984
Portugal . . . . .	152	766	645	434	264
Sierra Leone . . . . .	10				<sup>b</sup>
United States . . . . .	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>
<b>Rutile:</b>					
Brazil <sup>c</sup> . . . . .		35	96	116	287
Cameroun, French <sup>c</sup> . . . . .					45
Norway . . . . .	21 <sup>e</sup>	30 <sup>e</sup>	56 <sup>e</sup>	247	124 <sup>e</sup>
United States . . . . .	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>	<sup>d</sup>

<sup>a</sup> Compiled by Miss M. T. Latus, U. S. Bureau of Mines.

<sup>b</sup> Data not available.

<sup>c</sup> Exports.

<sup>d</sup> Bureau of Mines not at liberty to publish figures.

<sup>e</sup> Concentrates.

## IMPORTS AND CONSUMPTION

Beginning with 22,298 tons in 1930, and increasing to 142,740 long tons in 1936, the United States imported in the years 1930–1936, inclusive, 454,577 long tons of ilmenite. In the same period rutile imports grew from 3 to 510 short tons and a total of 1046 short tons. Declared values were \$6.75 per long ton for ilmenite in 1930 and \$4.19 in 1936. These figures, if correct, must have been prices at source. Rutile was declared at 14.5¢ per pound in 1930, at 3.1¢ in 1935, and at 3.78¢ in 1936.

The imports of ilmenite are somewhat larger than the consumption. The consumption of rutile during the period has been several times the imports.

## QUALITY OF ORES

Rutile usually carries, as marketed, 92.5 to 98 per cent  $\text{TiO}_2$ . Beach ilmenites carry between 51 and 60.3 per cent  $\text{TiO}_2$ . Norwegian ores have carried from 36.88 to 43.88 per cent  $\text{TiO}_2$ , as shipped.

The analyses of marketed ilmenite concentrates in Table 2 have been gathered from various authoritative sources.

TABLE 2.—*Comparative Analyses of Standard Ilmenite Ores*

Con- stituent	Piney River		American Rutile Co.	Travancore			Senegal	Norway		Malay States, Sample Shipments		
				Quilon	M. K.							
TiO <sub>2</sub>	44 28	42 64	51.41	60 3 <sup>a</sup>	54 3 <sup>a</sup>	53 56	54 71	36 98	43.88	51 70	54 00	52.52
FeO.	35 86	35 08	37.86	9 7	26 0	26 71	7 09	32.62	36.02	38.52	35 35	34.85
Fe <sub>2</sub> O <sub>3</sub> .	13 80	15.94	1.54	24.8	15.5	14.17	30 16	22 99	11.04	3.77	5 76	6.72
SiO <sub>2</sub> .	2 00	3 87	4 58	1.4	1.4	1 53	1 31	0.62	3.28	1.00	none	0.50
Al <sub>2</sub> O <sub>3</sub> ..	1.21		0 55	n.d.	n.d.	1.11	0 50	0.59	0 85			1.56
P <sub>2</sub> O <sub>5</sub>	1.01	0.67	0 17	0.17	0.26	0.20	0 14	0 06	0 02	0.09		
ZrO <sub>2</sub> ..	n.d. <sup>b</sup>			n.d.	n.d.	n.d.	2 37	none		none		
MgO.	0 07		2.35	n.d.	n.d.	1.01	0 89	3 04	3.67	0.21		
MnO.	0.52		0.70	n.d.	n.d.	0.38	1.32	0.25	0 33	3.15	3.95	3.40
CaO...	1.15		0 59	n.d.	n.d.	none	0.10	0 10	0 18	none		
SO <sub>4</sub> ...	n.d.						n.d.	0.40		0 05		
V <sub>2</sub> O <sub>5</sub> ...	0.16		0.07	0.26	0 20	0 028	0.274	0 476	0 20	0 04		
Cr <sub>2</sub> O <sub>3</sub> .	0.07		none	0.14	0.07	0.086	0.233	0.065	0 05	0.02	traces	
Na <sub>2</sub> O..										2.10		
Cl.....										n.d.		
Loss on ignition..	0 08			n.d.	n.d.	n.d.	0.92	n.d.				

<sup>a</sup> Average of shipments in 1935.

<sup>b</sup> Not determined.

## USES

As the pure or approximately pure metal, titanium has only small use. It has been made by heating the chloride with sodium and the oxide with calcium in bombs. Titanium of only 75 per cent purity has been made in a small way, and titanium of 96 to 98 per cent purity is quoted at \$6 per pound in trade journals. Such metal can be used in making the high-speed steel invented by Braid (U. S. Pat. 1955529), in which titanium replaces vanadium.

\* For a careful covering of the metallurgical uses of titanium see P. M. Tyler.<sup>47</sup> Free use has been made here of his article.

Ferrocabon titanium, made in the electric furnace, is produced in qualities: (1) containing 15 to 18 per cent Ti and 6 to 8 per cent C, sold at \$137.50 per ton; (2) with 18 to 20 per cent Ti and 4 to 5 per cent C, sold at \$142.50 per ton; and (3) with somewhat less carbon and some silicon sold at 20¢ to 25¢ per pound. Practically carbon-free ferrotitanium reduced by the thermit process, therefore containing 5 or 6 per cent Al, sells for about 32¢ per pound. Both types of ferrotitanium are used to deoxidize steel and to make closer grained cast iron.

The addition of 5 to 7 times as much titanium as the carbon contained in high-chromium steels takes the carbon from the chromium and makes the steel more easily rolled while hot, prevents air-hardening, makes it softer and more ductile after rolling<sup>4</sup> and prevents intergranular corrosion.

The making of a pure white titanium oxide from minerals containing at their purest between 30 and 50 per cent FeO is one of the most precise of chemical operations, and firms with seemingly ample financial and mechanical equipment, and with a staff of high order, have failed through long periods to conquer the technique. Processes are covered by hundreds of patents.

The United States makes most of the titanium pigments, and four companies are now conducting six factories. England, France, Italy, Czechoslovakia, Norway, Germany, Russia and Australia have two each and Japan has four.

The titanium pigment is made as pure titanium oxide, as titanium oxide with simultaneously precipitated barium sulphate, and as intimate mixtures of titanium oxide, and calcium sulphate. The pigments are used in nearly all American makes of enamel paints for inside use, and replace part of the other pigments in most good ready mixed outside house paints.

The pigments are also used in face powders and creams, paper, soap, linoleum, in artificial silk (to remove luster), in ground mica (to improve its color), in rubber, and in drawing and printer's inks. The making of titanium white is a growing business.

Rutile long had a very moderate use in enamels, in making ferrotitanium and a few titanium chemicals, but recent practice has shown that as a coating on rods to be used in electric welding it stabilizes the arc and a much more homogeneous and purer weld metal is deposited. In 1934, 15,000 tons of coated welding rod was sold,<sup>5</sup> on which 2000 tons of coating material was used, 10 per cent of which was probably rutile. In 1935 it was estimated by dealers that 1200 tons or more of rutile was used on welding rod. Ticasil, a mixture of rutile, lime and quartz, has been used as a substitute.

Rutile is also used in the manufacture of titanium tetrachloride for making smoke clouds to cover war movements and for use in sky-writing, but the tetrachloride is now made from ilmenite as a step in one method

of production of titanium white, so that rutile may no longer be necessary for that purpose.

Nearly all ilmenites carry some vanadium, generally less than 0.5 per cent, but reaching 5.51 per cent  $V_2O_5$ , or more, at Dublera, India,<sup>46a</sup> and the recovery of the vanadium has frequently been seriously proposed. Large-scale experiments have been conducted in Swedish steelworks<sup>48a</sup> and in 1932 a plant was erected at Kertsch, in the Crimea, in which "ferrotitanium and vanadium" were made from iron slags "making the Soviet Union independent of foreign sources."<sup>38a</sup>

In the United States, it was found, after careful research, that, although methods of extraction are technically practicable, vanadium can be obtained more cheaply from other sources.

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## CHAPTER XLVIII

### TRIPOLI

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THE word "tripoli" has been the general name for a number of comparatively soft, porous, friable and microcrystalline silicas of sedimentary origin. They are the end member of the chalcedonic group, opal being the other. They may contain traces of the  $\text{SiO}_2\text{-H}_2\text{O}$  system, the composition of "tripolite," a variety of diatomaceous earth which they may resemble somewhat in appearance as well as in name. A rock first exploited in 1871, near Dayton settlement, later called Racine and Seneca, Newton County, Missouri, was named tripoli and at later dates minerals of similar properties were also grouped under this name.

The consumers generally call the mineral from the Missouri-Oklahoma district tripoli, and minerals in the same group from other districts are known as "soft silica." Other names often used are "amorphous silica," "decomposed silica," and "cryptocrystalline silica." The name "soft silica" distinguishes such minerals from the very finely pulverized products made from "quartz sand" or "glass sand," for the ceramic trade.

The U. S. Bureau of Mines Minerals Yearbook groups all of the soft silicas and tripoli as "natural silica abrasives" and under the name tripoli includes rottenstone from Pennsylvania and a silica from San Bernardino, Calif. The so-called rottenstone is "a black impure shaly limestone grading into black shale" and is used as a filter. The exploited silica from Inyo and San Bernardino Counties, California, is of volcanic origin. Many textbooks and reference books of recent issue list the diatomaceous earths as tripoli. Diatomite appears to be the generally accepted term for the diatomaceous earths which are of organic origin and contain siliceous skeletons of microscopic aquatic plants known as diatoms and at times the siliceous skeletons of microscopic aquatic animal life.

#### COMPOSITION AND PROPERTIES

*Chemical Composition.*—Pure tripoli or soft silica would be 100 per cent by weight silicon dioxide ( $\text{SiO}_2$ ). The commercial products always contain associated minerals totaling a few tenths to several per cent by weight, with the silica content seldom less than 95 per cent. These associated minerals are not to be considered impurities because they

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\* Consulting Metallurgist, Joplin, Missouri.



impart certain desirable physical properties demanded by the consumers, just as pure iron with less than 1 per cent carbon, manganese, or combinations of elements added, may give definite new physical properties to the steel. These associated minerals are not part of the silica, but are surface coatings or partly fill openings between silica grains or fracture surfaces, and may be the principal cementing agency. Uncombined water is frequently present by reason of the large surface exposed per unit of mass.

Table 1 shows the chemical composition of commercial tripoli and soft silica and each mining district can produce mineral that will differ materially from the figures shown.

TABLE 1.—*Chemical Composition of Tripoli and Soft Silica<sup>a</sup>*

Constituent	Composition, Per Cent					
	Illinois	Arkansas	West Tenn. Valley	East Tenn., Georgia	Missouri- Oklahoma "Cream"	"Rose"
SiO <sub>2</sub> . . . . .	99.44	99.72	98.92	93 to 98	97.70	98.00
Al <sub>2</sub> O <sub>3</sub> . . . . .	0.22	0.067	0.46	1.0-1.4	0.838	0.19
Fe <sub>2</sub> O <sub>3</sub> . . . . .	0.14	0.003	0.07	0.22-1.6	0.282	0.73
CaO . . . . .		0.090	0.37	Tr-0.30	0.001	Tr
MgO . . . . .		0.001		Tr-0.20	0.014	Tr
Na <sub>2</sub> O-K <sub>2</sub> O . . . . .					0.148	0.29
TiO <sub>2</sub> . . . . .				Tr-0.20	0.059	
MnO . . . . .					0.002	
H <sub>2</sub> O (clay) . . . . .					0.099	
Ignition . . . . .	0.25	0.120		0.3-0.7	0.501	0.50

<sup>a</sup> The chemical analyses of the Missouri-Oklahoma tripoli are by C. V. Millar, Chemist, Joplin, Mo. The other figures are from producers in their districts.

*Physical Properties.*—Tripoli and the soft silicas when very finely pulverized and when tested in a pycnometer, show a specific gravity as high as quartz (2.654). The apparent specific gravity of a mass obtained from the dry weight and weight in a liquid varies with the size of sample and of the individual fragments because of the "air-locked" spaces not filled by the liquid, and the determinations indicate from 2.00 to 2.50. When the weight per unit volume is found by mercury displacement, the calculated weight per unit may be as low as an equivalent volume of water.

The Georgia-Tennessee and western Tennessee Valley varieties are loosely coherent masses of individual grains of silica some 2 microns in diameter. The denser and more firmly consolidated Illinois phase is a much more coherent aggregate of individual grains with an average diameter of less than 2 microns. The Arkansas variety does not break down into individual silica grains, as the sharp-edged fragments of this decomposed chert when water-ground will produce a soft, smooth talclike material, the individual particles being of smaller diameter than the

Illinois phase. The Missouri-Oklahoma tripoli varies from a loose unconsolidated mass to the firm coherent grade that is carefully selected and processed for the market. These commercial grades contain very little hard, free quartz, chert or flint grains, but each particle produced in grain-size reduction remains a fibrous, porous structure of silica, as is shown when the surfaces of the smallest grains are studied at some 2500 diameters. This property distinguishes tripoli from soft silica.

All exploited deposits contain material that varies in color from white to deep hematite red. A Georgia deposit shows soft yellow and light orange tints. A pink cast is noted in Arkansas and the Missouri-Oklahoma variety is never found as white as other deposits, and is termed "colored tripoli" by the ceramic and pigment trade, because the color varies from a light cream to a deep rose. However, it is possible to decolorize this mineral for special purposes.

Most of the varieties of soft silica, except the Illinois hardest rock, can be cut by the fingernail, and small fragments can be reduced in grain size by rubbing between the fingers. The feel of the finely pulverized minerals between the fingers varies from the characteristic "harsh" feel of the Missouri-Oklahoma variety to the soft talclike feel of the Arkansas phase.

As the dry weight per unit volume varies, so will the interstices vary, but not necessarily in direct proportion; so that when a specimen is submerged in water the percentage of voids remaining unfilled may vary greatly for several specimens. This measure of the porosity of a mass of tripoli has a distinct value for gauging the value of the pulverized product to be made from it, especially when buffing and polishing products are involved. The Missouri-Oklahoma tripoli is the most porous of the firmly consolidated silicas. For example, a cube of cream tripoli with an edge of 3 in., thoroughly oven-dried, plunged in water and submerged, may increase in weight more than 50 per cent in less than one minute. A dense, fast-buffing grade rose tripoli block of the same size when tested in a like manner may increase in weight less than 25 per cent and more than one hour may be required to gain a constant weight.

Some of these silicas, when wetted and dried repeatedly, tend to become more firmly consolidated, the Missouri-Oklahoma variety being the best example. When wet in the quarry, the material can be cut into building blocks, and after exposure to the air becomes very firm. Blocks of tripoli have been used for building purposes, and a house more than 50 years old and porch pillars more than 30 years old today appear to be as well consolidated as fine-grained sandstone.

The less firmly consolidated silicas disintegrate when weathered, and the apparently firm, white, altered chert of Arkansas, as it weathers, disintegrates into small sharp-edged fragments.

The hardness scale of all of these silicas is comparatively low, but as

the mineral is reduced in grain size the smallest individual particles have a hardness approaching that of quartz crystals. Because of its high porosity the Missouri-Oklahoma tripoli has a unique characteristic when used as a buffing or polishing agent. It sticks to the buff better than the smooth-faced and sharp-edged chert, quartz and flint particles that are thrown off by the centrifugal force; also the hardness of the grains increases as the size of the grains decreases. All of the whiter soft silicas and the lighter colored tripoli, when pulverized dry, become supercharged with static electrical charges, and sizing difficulties may result.

In general, the crushed particles of these minerals when smaller than 30 microns are comparatively hard, coherent aggregates of finer grains of rounded and botryoidal or incrustated forms, the Missouri-Oklahoma tripoli being the least dense and the Arkansas mineral the finest grained.

#### ORIGIN AND MODE OF OCCURRENCE

The soft silicas of Illinois, Tennessee, Georgia, Alabama and Mississippi are generally assumed to be the residual products of the decomposition of a highly siliceous limestone or dolomite, the carbonate having been dissolved by the circulating solutions, leaving the grainy-type silica skeleton. The Arkansas variety appears to be the product of disintegration or alteration of a chert by weathering. The Missouri-Oklahoma tripoli being different from these others, appears to have had a distinct mode of origin. A study of chertification in the Tri-State zinc district by G. M. Fowler and associates,<sup>5</sup> indicates to them that tripoli has been formed in the same manner as the Illinois-Tennessee soft silicas, for they assume that all highly siliceous and very porous rocks having a higher percentage of silica than their "cotton rock" are to be called tripoli. H. S. McQueen,<sup>11</sup> of the Missouri Geological Survey, developed a technique for dissolving limestone and dolomite by hydrochloric acid and from the resultant residues can identify geologic formations. He says (private communication) that some residues from highly siliceous limestones appear to resemble tripoli. Long-range observations and casual study of the Missouri-Oklahoma tripoli beds parrot the general literature, but detailed study by qualified geologists and mineralogists has indicated to them that the generally accepted theory may not be the true solution of the problem of how this particular silica was formed. Cox, Gottschalk and Dean<sup>3</sup> supported the gel theory of siliceous mineralization to account for the tripoli in the Tri-State district, and later Levings postulated a theory that merits consideration, in view of the fact that he has made a definite study of the tripoli deposits for many years.

These deposits may have been laid down in essentially their present form, as a flocculant colloidal silica, together with alkaline salts that

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<sup>5</sup> References are at the end of the chapter.

were later leached away by circulating solutions; or they may have been the result of recrystallization of a colloidal silica. The fibrous, porous and flowlike structure of this tripoli, examined at some 2500 diameters, when contrasted with cotton rock, the so-called tripoli in the zinc-field chert beds, and the tripoli-like masses observed in certain insoluble residues, lessens one's belief in the idea that all of the soft silicas and tripoli have had a common origin.

The Illinois and western Tennessee Valley silica is generally horizontally bedded with definite stratifications. The Tennessee-Georgia-Alabama beds of commercial silica are confined between lateral side walls with a dip of about 45° or less. The Arkansas silica deposit being operated is a 20-ft. seam on the flank of a low mountain, the material becoming harder in the roof and floor and in the face as the drifts leave the outcrop. The Missouri-Oklahoma tripoli is found in horizontal beds some 10 ft. thick, as an average, within a few feet of the surface of the tops of hills or ridges. The stratification is usually horizontal, but rolls and folds occur, with fracture seams at various angles from horizontal to vertical.

#### DISTRIBUTION OF DEPOSITS, PRODUCTION AND CONSUMPTION

There are but five districts of commercial soft silica and tripoli in production today (1937):

1. The Missouri-Oklahoma district is a small area roughly outlined by Racine and Seneca, in Newton County, Missouri, and Peoria, in Ottawa County, Oklahoma.

2. The Illinois district is in the southern part of the state, Alexander and Union Counties.

3. The Tennessee-Georgia-Alabama district includes several counties in each of the three contiguous states.

4. The western Tennessee Valley district includes the area where the three states, Tennessee, Alabama and Mississippi, join.

5. The Arkansas producer mines the mineral about six miles from Rogers, in Benton County, and mills it in Rogers.

Tripoli and soft silicas have also been reported in Indiana, Iowa, Kentucky and Minnesota. Very fine-grained silica, called tripoli, has been reported in many parts of the world; all appear to be of the same classification as the Illinois-Tennessee type, but there is no definite information of any deposit ever being found with the mineral having the same distinctive properties as the Missouri-Oklahoma tripoli.

*Production and Consumption.*—Production figures for individual producers cannot be revealed, but the Illinois and Missouri-Oklahoma districts are the principal producers. The industry is not large, but because of the great care exercised in mining and processing, the tonnage of finished material per man-day is small and the industry supports many

families. The United States Bureau of Mines 1937 Minerals Yearbook (p. 1286) reports the following figures for 1936:

	Total Tons	Total Value	
		Crude	Crude and Finished
Illinois . . .	10,981	\$21,962	\$138,063
Other states	17,506	61,546	253,815
	28,487	\$83,508	\$391,878

#### POLITICAL AND COMMERCIAL CONTROL

The United States is independent of the world for the soft silica and tripoli demanded by the trade today. It exports the Missouri-Oklahoma tripoli, pulverized and of definite gradation, for the buffing and polishing trades. England imports the largest tonnage, and direct shipments are made to France, Holland, Germany and other European countries. Tripoli is also sent to Canada, India, Far East ports and to South America. As there are fewer than three producers of this mineral product, no accurate figures may be published, but it is assumed that several thousand tons are exported each year.

#### PROSPECTING, EXPLORATION AND MINING

The most desirable commercial grades of tripoli in the Missouri-Oklahoma district usually are covered with a light overburden. Where outcrops are clean-faced, the tripoli is usually too dense or firmly consolidated. Test pits are dug by hand on the crests of ridges and tops of the low hills in "wildcat" manner. If more than 6 or 8 ft. of overburden is passed through before "rotten tripoli" is found, the pit is abandoned unless other pits have indicated that good commercial tripoli may be found at a greater depth. The overburden is usually an unconsolidated mixture of chert boulders and fragments with gravel, sand, clay and soil; and next to the tripoli bed lies the rotten tripoli, an unconsolidated formation of true tripoli and red clay minerals. When sufficient test pits have proved a commercial bed of tripoli, the underbrush and trees are removed, a drain is cut, and the overburden is removed by power shovels.

The exploited silicas of the Tennessee-Georgia field are studied from outcrops. In developing a Georgia property, the surface matter is removed, and the top and the face of the deposit are cleaned. The quarries are opened in benches and the silica is removed and sorted for color. Other silica deposits are studied from outcrops and by driving tunnels into the face; prospect drifts are cut and the face, roof and floor are studied by drilling with coal augers or core drills.

*Mining Methods.*—The methods of mining in the southern Illinois field and western Tennessee Valley district are much alike. A modified room-and-pillar system is used, but because of lack of consolidation of the mineral, both rooms and pillars have about the same area. In Arkansas the mine has a shaft sunk on the side of the mountain about 50 ft. from the outcrop and through some 30 ft. of unworkable strata into the commercial grade of silica. Tunnel openings are usually timbered, and the rooms have a dome-type roof to better support the shattered rock. Drilling is done by both hand-steel and machines, depending upon the hardness of the rock. Blasting is by slow dynamite and black powder, like that used by coal miners. Much experience is required to drill a round effectively and load and shoot any of the silicas, as a slight overcharge of powder will merely chamber a hole and break little tonnage. No special drilling pattern is followed, as the better results are obtained by following previous experience of changing conditions.

Boulders are broken to one-man size by the use of splitting wedges and sledges. The broken rock is hand-loaded into tramcars and sent to the mill in various ways. At the Arkansas mine the broken rock is loaded into Tri-State zinc district "cans," hoisted and dumped into a hopper, then hauled by trucks to the mill some 7 miles away.

The Georgia property being an open quarry, the desired grade of silica is shot down, sorted for color and dried; the greater portion of the tonnage being sent out of the district as untreated silica.

All of the mining in Missouri and Oklahoma is by simple quarry operation. At one time large quantities of even-textured tripoli were carefully quarried and sorted to be made into filter stones, and no explosive was used to work the quarry. In 1937, some filter stones were still being made for export. The major tonnage of rock from the quarries is classed as "crush," and is sorted into three general groups according to color: white, cream and rose. Vertical holes are spaced at irregular intervals and are drilled from 3 to more than 10 ft. deep. Lifter holes are used at times, and if soft horizontal seams occur the face may be benched. The less uniform the color, the greater the care there must be in shooting, as the least amount of fines is desired so that the workmen can sort with the least effort. Black powder or slow modified dynamite is used.

#### PREPARATION FOR MARKET

The crude mineral from quarry or mine is carefully sorted for color and texture and air-dried for shipment as "crude rock." Most of the silicas require several weeks to air-dry when they are to be dry-processed near the mine or quarry. The Missouri-Oklahoma tripoli may require several months to reach an equilibrium with the atmosphere.

Grinding and sizing methods used in the milling operation are either dry, wet, or combinations of the two. Each method has merit, and the

method used in each mill has been adopted because apparently it best fits the particular problem. The preparation of the soft silicas and tripoli is a comparatively simple operation; that is, grain-size reduction. However, the details of controlling milling procedure and producing uniform products to meet the increasingly difficult specifications of the buyers of these minerals complicates the milling operation. The trade may demand superfines, uniform grain size, limits for larger grains, and gradations of the sizes to give a certain oil-absorption number; and the modern mill flowsheet must be sufficiently flexible to make the products ordered.

One wet-grinding mill in Illinois, treating a very white silica, crushes the mine product to pass a  $\frac{1}{2}$ -in. screen. This product is ground in a tube mill, silex-lined, using flint pebbles. The mill works in closed circuit, with bowl classifiers and with milling conditions established to make two distinct products for the pigment trade, a very fine-grained and a superfine grained product. A mill in southern Illinois, wet-grinding in a like manner, makes seven classes of finished material, which grade from 65 to 99.9 per cent passing the No. 325 testing sieve. A dry grinding process with air classification is also used.

A mill in Arkansas grinds wet, but the finely ground product after being dried may be reclassified by an air separator to provide a special product. A pebble-lined tube mill is charged with raw rock, coarsely crushed, the larger pieces being about 6 in. in diameter. The grinding characteristics of this silica are distinctive, because it is a badly fractured, decomposed chert, and the larger pieces make ideal grinding media when established pulp densities are used. The product from the tube mill is carefully water-classified by using settling cones in series, and the cone products desired are thickened and dried in a steam-heated rotary drier. The final water overflow is carefully regulated to remove from the circuit much of the highly colored colloidlike minerals that stain the fracture surfaces of the mine rock. Milling conditions have been established to make the three principal products, all of them passing the No. 325 sieve and marked: (1) admix 025, (2) white 325-400, and (3) float 600. All three products have about 60 per cent by weight of the individual grains smaller than 10 microns in diameter.

A mill in the western Tennessee Valley district predries the mine rock and pulverizes the easily crushed material in a 6 by 2-ft. Hardinge pebble mill, operating in closed circuit with an 8-ft. Gayco air separator. The mine-run material is air-dried to about 9 per cent moisture before being sent to the mill circuit. As the silica is noncoherent, the pebble mill is more of a disintegrator than a grinding machine, and the discharged product is screened at  $\frac{1}{4}$  in. to remove unweathered chert fragments before being sent into the air-separator circuit. Four gradations as to grain size are reported: "60 mesh," 65 per cent passing 200; "80 mesh,"

85 per cent passing 200; "100 mesh," 95 per cent passing 200; and "200 mesh," 85 per cent passing the No. 325 testing sieve.

Other mills, some of which are inactive at present, when milling the soft silica minerals, follow in general the flowsheets outlined in the preceding statements.

The preparation of Missouri-Oklahoma tripoli differs from that of the soft silicas. The deeply stained coherent mineral is demanded by the buffing trade, and the lighter tinted tripoli is prepared for the polishing and foundry-parting markets, a very small tonnage being used by the pigment and ceramic industries. There are but two mills preparing Missouri-Oklahoma tripoli for the market. Both use the dry grinding process, and each follows the general milling procedure developed to meet the special demands of the trades.

The raw quarry rock is hand-sorted for color and texture into three general groups. It is ricked in air-drying sheds for about six months before being sent to the mill. At the mill it is coarse-crushed by hammer mill or a coal breaker and smooth-face rolls to pass about  $\frac{1}{4}$  in. Each mill has a rotary tube drier about 50 ft. long and 7 ft. in diameter, heated with wood or oil burners. The dried material is pulverized in a Hardinge mill or tube mill, which operates in closed circuit with Hummer vibrating screens or a mechanical vibrating screen manufactured locally. The grinding, conveying and screening units operate under reduced atmospheric pressure, and the driers have a regulated draft for the collection of dust. Exhaust fans are placed next to baghouses where the dust is collected. Between the exhaust fans and the source of dust, cyclone-type cones are in the train for the removal of the larger particles of dust.

In general, the milling is simple, but the exacting specifications of the buyers demand that the milling flowsheet be flexible in operation. As each unit must be easily controlled, a well developed technique has been established for the several grades of finished tripoli to be prepared. No mineral is added to or taken from the mill feed, and the finished product is classified by color and grain size. The darker tinted tripoli is less difficult to prepare, but special precautions are required in making some of the products from the lighter tinted tripoli, because the fine particles become charged with static electricity under certain conditions, and screening becomes difficult to control. Although three classes of colors and but four general screen-sized products are produced, combinations of these factors demanded by the trades in the past 50 years have increased milling costs.

#### TESTS AND SPECIFICATIONS

Unfortunately, there are no standardized specifications or methods of testing procedure for most of the finished silica and tripoli products.



Color comparisons are made by matching in water or paint vehicles with previous shipments or a "standard" arbitrarily established. Sieve sizes are mentioned for maximum grain size tolerated and figures of the weights per cent passing or retained on sieves of smaller openings. Almost all the tests are empirical, and of two products for the pigment trade, both passing the No. 325 sieve, when "rubbed out" with a light oil on polished glass, with a steel spatula, one may be more "gritty" than the other and not pass the inspector. Two finished buffing grades of rose tripoli may have identical screen analyses, yet the oil adsorption may differ because the subsieve size-frequency curves are different. The Scott paint volumeter is used at times to establish loose, dry, bulk weight, and settling tests are made by shaking a given weight in a volume of water and noting the time for the water to clear to a given depth. Lately, more thought is being given to the determination of the subsieve sizes, for they have a definite bearing on the physical properties of these finely pulverized minerals. A mill at Racine, Mo., makes use of this test as a milling control, a rapid technique having been developed to make six readings between 40 and 20-micron sizes in less than 10 minutes. When these data are plotted on log-log graph paper, the straight line indicates weight-percentage figures for grain sizes less than 5 microns in diameter.

The Missouri-Oklahoma tripoli is roughly grouped as to grain size as follows: (1) air-float—all passing the No. 200 sieve and 98 per cent passing the No. 325; (2) parting—98 to 99 per cent passing the No. 200 sieve; (3) admix—90 per cent passing the No. 200 and 30 per cent smaller than 10 microns; (4) buffing—minus 100 per cent passing to No. 40, and 80 to 85 per cent passing the No. 200; (5) extra fine admix—70 per cent smaller than 10 microns.

#### MARKETING, USES AND PRICES

The finished products are sold direct to consumers and also to brokers or trade-supply houses for local distribution. The crude minerals are sold direct to grinders and other users. The finished products are usually packed in paper or paper-lined jute sacks. At times, special packings for export or for the special requirements of buyers are used. The weight per unit ranges from 50 lb. to more than 200 lb., net. Rail shipments are made in closed cars of not less than 30 tons net.

Most of the producers of the finished products, having been in business for many years, have established contact with regular customers. These buyers are very careful about making any changes in procuring their supplies and may hesitate between the products of two mills making the same class of material from raw mineral in adjacent deposits, because the range of the specifications submitted is insufficient to predict the

results that will be obtained in practice. The distribution of sales of the minerals classed as tripoli is given in Table 2.

TABLE 2.—*Tripoli Sold or Used by Producers in the United States in 1936<sup>a</sup>*

Use	Number of Producers Reporting	Short Tons	Value as Sold (Crude and Finished)	Percentage of Total Accounted for	
				Quantity	Value
Abrasives.....	5	16,943	\$247,948	59.8	64.0
Concrete admixture.....	3	2,074	27,834	7.8	7.2
Filler.....	4	3,107	38,454	11.0	9.9
Foundry facing.....	2	b	b		
Miscellaneous.....	3	6,213	73,142	21.9	18.9
Totals accounted for . .	7 <sup>c</sup>	28,337	\$387,378	100.0	100.0
Use not specified . . .	1	150	4,500		
Grand total.. . . .	8	28,487	\$391,878		

<sup>a</sup> Minerals Yearbook, 1937, U. S. Bureau of Mines.

<sup>b</sup> Included under "miscellaneous."

<sup>c</sup> A producer reporting more than one use is counted only once in arriving at total.

The price received for crude silica of the general classification indicated has been as low as \$3 per ton at the mine. The selected grades of crude sell for as much as \$8 per ton on the car. The prices received for finished products fluctuate slowly. Quality of product, even at a higher cost, is demanded by the consumer rather than an inferior product that could be produced at a lower cost on a tonnage basis. In the early part of 1937, producers reported prices for finished products as follows:

Illinois and Tennessee . . . . . \$3 to \$40 f.o.b. mill, 30 tons net.  
 Arkansas..... \$3 to more than \$22.50 f.o.b. mill, 30 tons.  
 Missouri-Oklahoma..... \$15 to \$25 f.o.b. mill, 30 tons.

These prices are for the commercial grades usually demanded by the trades. However, special specifications are supplied at times at an advance in the prices quoted. The trade journals—*Metal Industry*, *Chemical Markets*, *Engineering and Mining Journal*, etc.—list these silicas and tripoli in their price columns for chemical and raw materials, which show the slow movement of prices through the years.

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